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PREVENTION, DETECTION, AND SUPPRESSION OF HYDROGEN EXPLOSIONS IN AEROSPACE VEHICLES

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by
G. J. Caras

March 1966

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Research Branch
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Redstone Arsenal, Alabama 35809

ABSTRACT

This report reviews and summarizes the hazards to aerospace vehicles caused by hydrogen. Topics such as flammability limits of hydrogen in hydrogen-oxygen and hydrogen-air mixtures, methods of detecting hazardous conditions, and means of inhibiting fires and explosions are discussed. Although relatively few references were found in the literature concerning hydrogen fire and explosion hazards in aerospace vehicles specifically, it is generally believed that many of the procedures used in the handling, storage, and transportation of liquid hydrogen in more conventional applications also apply to hydrogen used as a fuel in space vehicles.

FOREWORD

This report attempts to review and summarize the published literature concerning the hazards to which aerospace vehicles may be exposed because of the use of liquid hydrogen as a fuel. The literature search, which covered the period of 1955 to the present time, yielded relatively little information concerning the safety aspects involved in the use of hydrogen in space vehicles. This, of course, was not surprising since most of the technology that has been developed for the safe handling of liquid hydrogen in other applications also applies to aerospace vehicles.

The information for this report was taken from the sources available at the Redstone Scientific Information Center which, in addition to the Center's holdings, includes NASA and DDC computer tape searches.* The following indexes also were consulted for the time period covered in this report:

Applied Science and Technology Index
British Technology Index
Chemical Abstracts
Chemical Propulsion Abstracts
Engineering Index
Fuel Abstracts
Government-wide Index to Federal Research and
  Development Reports
Index Aeronauticus
Journal of Applied Chemistry Abstracts
Nuclear Science Abstracts
Science Abstracts

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*The NASA tape searches provide references from the Scientific and Technical Aerospace Reports (STAR) and the International Aerospace Abstracts, while DDC searches provide references to the report literature in the Department of Defense.

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Section I. INTRODUCTION

The primary impetus behind the production and use of large quantities of liquid hydrogen has been the development of upper stages for large space rockets. Liquid hydrogen, because of its high reactivity and the low molecular weight of its combustion products, produces more specific impulse than any other chemical fuel. These advantages, however, are accompanied by some disadvantages, one of which is the hazard associated with its use. Hydrogen gas, because of the small size and high mobility of its atoms, can leak through air-tight seals; and if it leaks into a confined space where air or oxygen is also present, it can form a detonable gas mixture. Prevention of such situations by proper design of aerospace vehicles is, of course, of utmost importance; but in spite of all the preventive measures, hazardous conditions still can be encountered.

A considerable amount of information is in the literature concerning the flammability and detonation properties of hydrogen and the chemical reaction between hydrogen and oxygen.[1,2] Although an understanding of the reaction mechanism between hydrogen and oxygen is a necessary part of any consideration of the hazardous characteristics of hydrogen, little such information is included in this report. Lewis and von Elbe have studied these properties extensively, and the reader wishing more information on this subject is referred to their work.[1] There was relatively little information in the literature concerning the suppression or inhibition of hydrogen-oxygen fires, and even less on the hydrogen hazards to which aerospace vehicles may be exposed.

Considerable effort is being made in the area of detection of hazardous hydrogen mixtures, and a large portion of this survey is devoted to this topic. It was found that developments in this area could be most conveniently summarized according to the organization by which the work was performed. Although every effort was made to include all significant work as reflected by the literature, the list of organizations working in this field may not be complete.

The last section of this survey, entitled "Hydrogen Hazards to Saturn Space Vehicles" is an attempt to summarize the current thinking concerning the hazards to which these space vehicles may be exposed because of their use of liquid hydrogen engines. It appears that most safety principles are based on proper design to prevent the formation of dangerous gas mixtures and/or their ignition, rather than to combat a fire or explosion after it is initiated. A portion of the information contained in this section was gathered from discussions with several persons at NASA's Marshall Space Flight Center.

Section II. LIMITS OF FLAMMABILITY AND DETONATION

1. General

The principal hazard associated with gaseous or liquid hydrogen is its ease of ignition over a wide flammability range in air and oxygen. The combustion limits of hydrogen in air range from about 4 percent to 74 percent hydrogen by volume. In oxygen the upper limit is extended to about 94 percent. The limiting compositions for detonation of hydrogen-air mixtures are from approximately 18 to 59 percent hydrogen by volume. Figure 1 gives the flammability limits of some common gases in air. Only hydrazine and acetylene have broader ranges of flammability than hydrogen.[3]

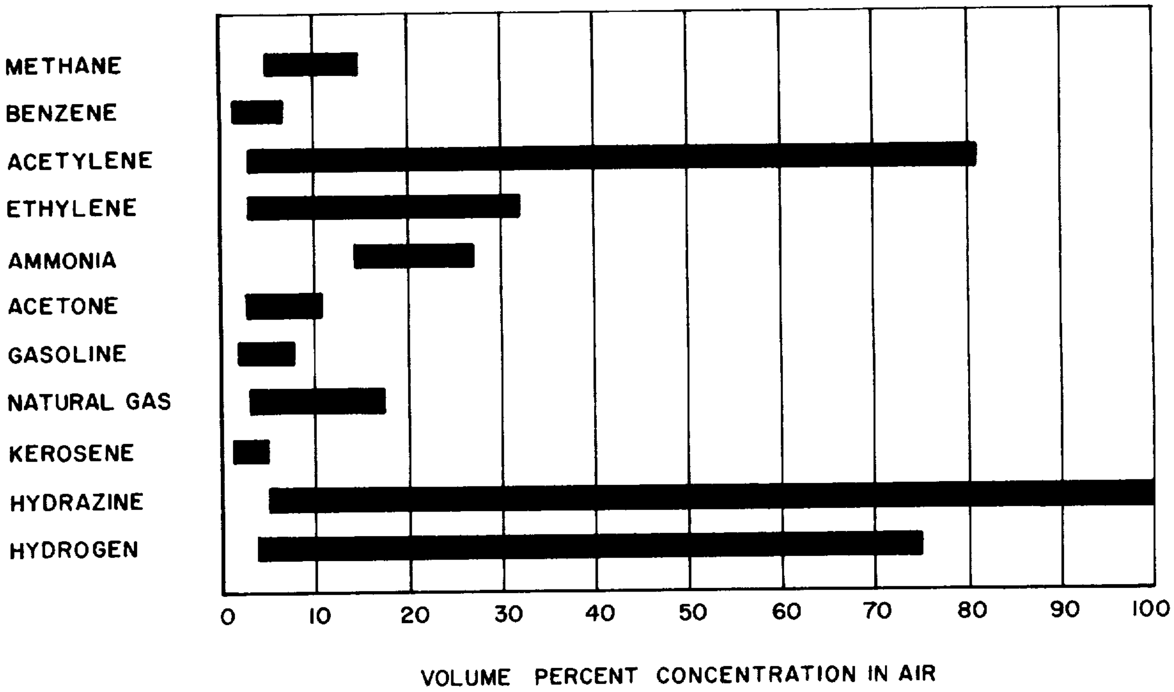

Figure 1. Flammable Limits in Air

The ignition temperature of hydrogen in air at atmospheric pressure is approximately 1075° F, while most hydrocarbons can be ignited between 400°and 600° F. This may lead one to believe that hydrocarbon fuels are easier to ignite than hydrogen, but actually the energy required to enflame hydrogen is only about one tenth of that required to enflame typical hydrocarbons. Figure 2 shows a comparison of the spark ignition energies of hydrogen-air and propane-air mixtures.

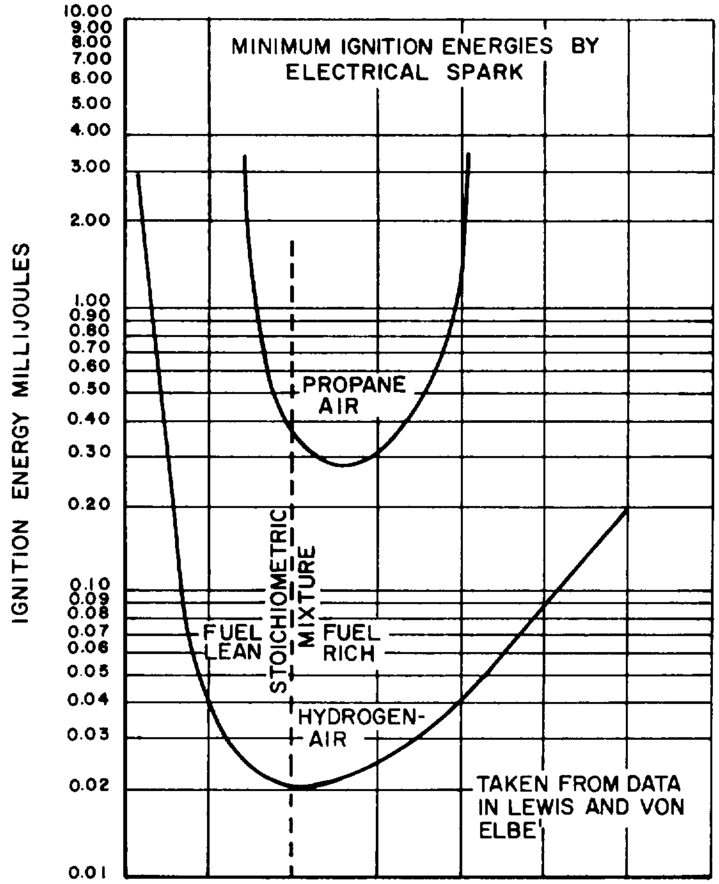

Figure 2. Spark Ignition Energies of Hydrogen-Air and Propane-Air Mixtures

Figure 3 is a plot of ignition energy against fuel concentration for various mixtures at atmospheric temperature and four different pressures. As the pressure is lowered, the ignition energy increases rapidly. It also appears that the minimum spark ignition energy occurs near the stoichiometric ratio of hydrogen and air regardless of pressure.

Suppressing a hydrogen flame is more difficult to achieve than for most hydrocarbons as indicated by the quenching distances in Figure 4. Quenching distance is defined as the distance between two parallel walls which will just permit a flame to pass without cooling it to extinction. The smaller the quenching distance, the greater the resistance of the flame to extinction.

The limits of detonation of hydrogen-air mixtures are approximately 18 to 59 percent hydrogen by volume. These detonation limits are of concern where possible confined conditions or strong detonating ignition sources exist. Experiments with hydrogen indicate that even partial confinement such as that produced by three vertical walls (roof and one side absent) is sufficient to produce a detonation if mixtures within the detonation limits are ignited.[4] Every effort should be made to avoid even partial confinement, particularly where large quantities of hydrogen are involved.

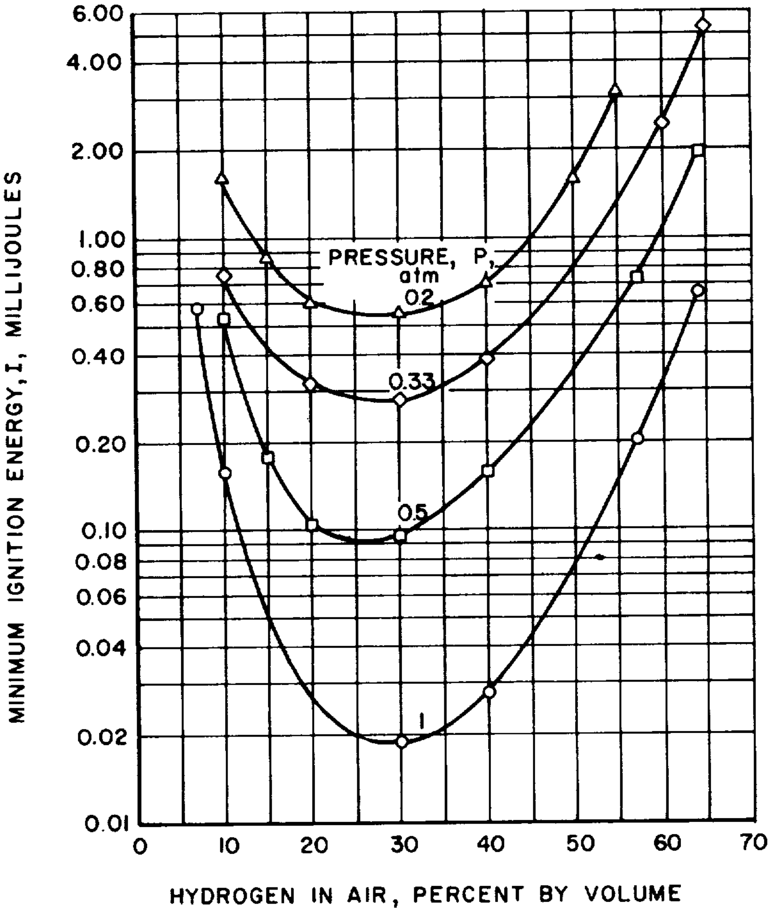

Figure 3. Spark Ignition Energies for Hydrogen-Air Mixtures at Various Pressures

The possibility of detonation in free space was studied by Cassutt, Maddocks, and Sawyer.[5] These investigators found that near-stoichiometric mixtures containing 32 percent hydrogen could propagate three-dimensional shock waves in free space if a sufficiently strong initiating source were available. One of the potential hazards associated with detonations in free space is the gross spillage of liquid hydrogen.

For example, the contents of a 60,000-pound storage tank would produce 2,100,000 pounds of detonable mixture if mixed with air in stoichiometric proportions.

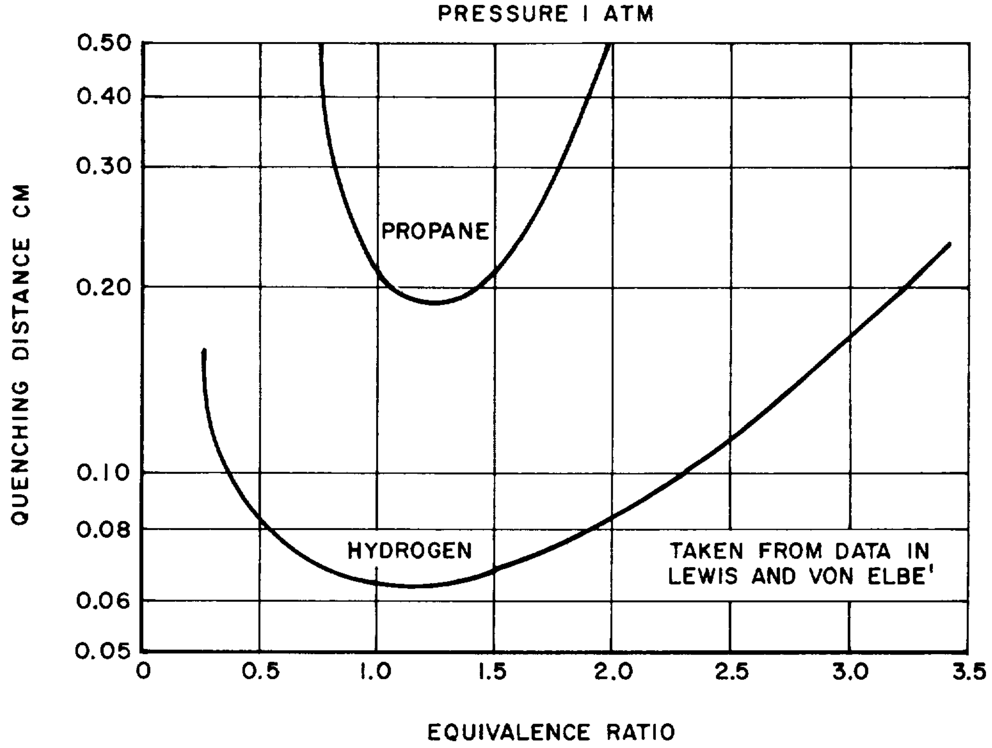

Figure 4. Quenching Distance for Hydrogen and Propane

A number of tests were carried out in which five- and eight-foot diameter latex balloons were filled with mixtures of hydrogen and air and initiated at the center with explosive and flame sources, hot wires, or sparks. A minimum initiating source of two grams of pentolite was required for full explosive yield. Use of a blasting cap initiator, which is approximately one-half gram of explosive, reduced the yield 95 percent, while flame sources and hot wires gave only combustion of the gases with no measurable pressures. It was concluded that the detonation of a hydrogen-air mixture in free space is possible only if a suitable mixture ratio and a sufficiently strong initiating source are present. Since the probability of these conditions occurring is remote, the chance of detonating a large mass of hydrogen in free space is quite low.

In summary, liquid hydrogen does not normally present an explosive hazard when it evaporates and mixes with air in an unconfined space. The explosive hazard exists when the mixture is confined, partially confined, or ignited with a shock source initiator such as TNT.

2. Influence of Pressure and Temperature on Limits

Figure 5 shows various results obtained for the limits of hydrogen in air under pressures greater than atmospheric. The differences are not so great as they may appear to be at first sight, except for lower-limit mixtures at pressures of one to five atmospheres, and these may be ascribed to different interpretations of experimental results rather than to the experiments themselves. The criterion was 100 percent combustion for the series indicated in the figure by small circles but only 80 percent for the series represented by the unbroken line. In general, it appears that the limits are at first narrowed by increase of pressure above atmospheric but at higher pressures are steadily widened.

Reduced pressure affected the lower limit as follows: In a closed glass tube six centimeters in diameter and 120 centimeters in length, with downward propagation of flame, at one atmosphere the lower limit was 9.35 percent and at one-half atmosphere, 10.6 percent.[6] Experiments in a burette indicated the existence of a small lowering of the lower limit between approximately 500 and 200 millimeters pressure followed by a rapid rise between 200 and 100 millimeters.[7] Other experiments in narrow tubes (8, 16, and 25 millimeter diameter), with upward propagation of flame, showed no great change in the limits until the pressure was below approximately 300 millimeters. When the range narrowed until below 50 millimeters, no mixture propagated flame.[8]

Table I and Figure 6 give some results obtained in a closed tube 2.5 centimeters in diameter and 150 centimeters in length, with downward propagation of flame.[9] The flame temperature necessary to insure propagation of flame downward is much above the ignition temperature of hydrogen in air, 585° C. Moreover, increase in initial temperature of the mixture does not cause the calculated flame temperature of the mixture to fall toward the ignition temperature but has the opposite effect for lower-limit mixtures.

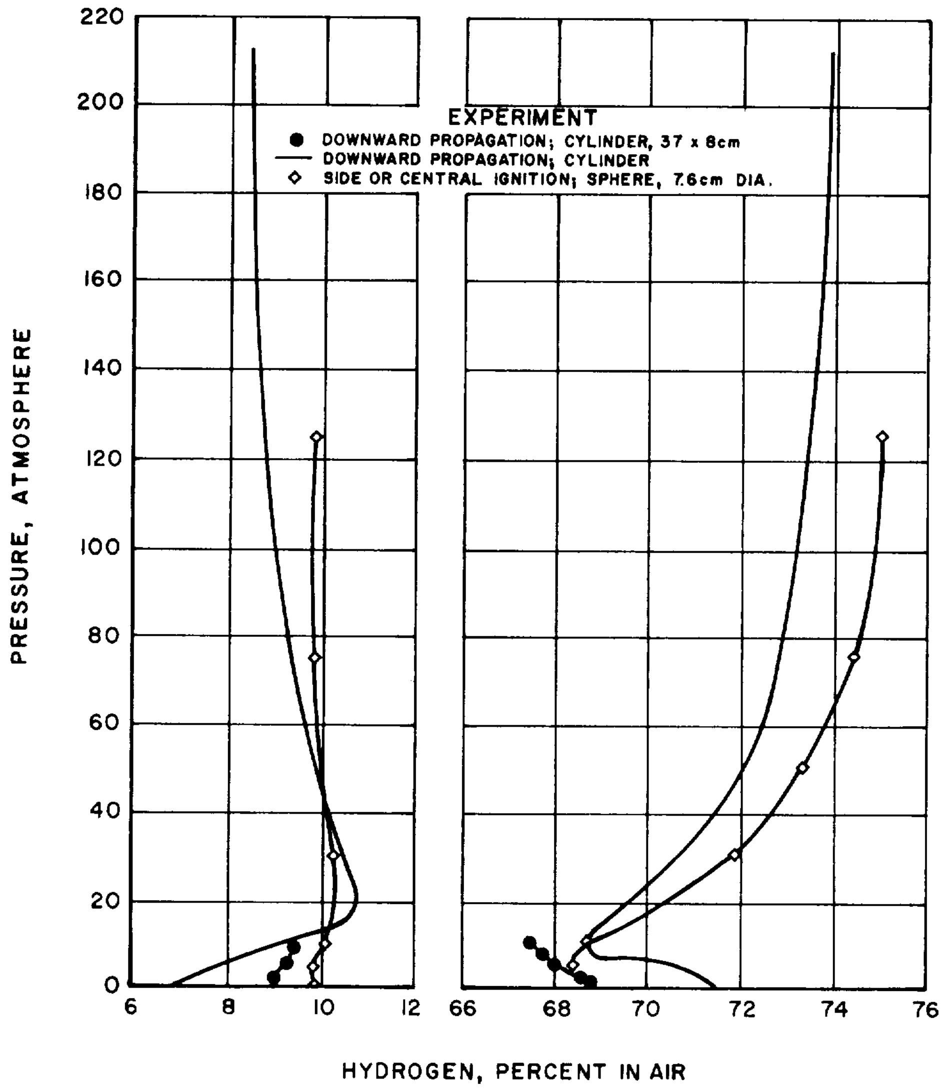

Figure 5. Effect of Pressure Above Normal on Limits of Hydrogen in Air

Table I. Influence of Temperature on the Limits of Flammability of Hydrogen in Air, Downward Propagation of Flame

Temperature, °C	Limits of Flammability, Percent Hydrogen		Calculated Flame Temperatures, °C	
	Lower	Higher	Lower Limit	Higher Limit
17 ± 3	9.4	71.5	815	980
50	9.2		820	
100	8.8	73.5	835	970
150	8.3		830	
200	7.9	76.0	845	975
250	7.5		860	
300	7.1	79.0	875	970
350	6.7		890	
400	6.3	81.5	900	980

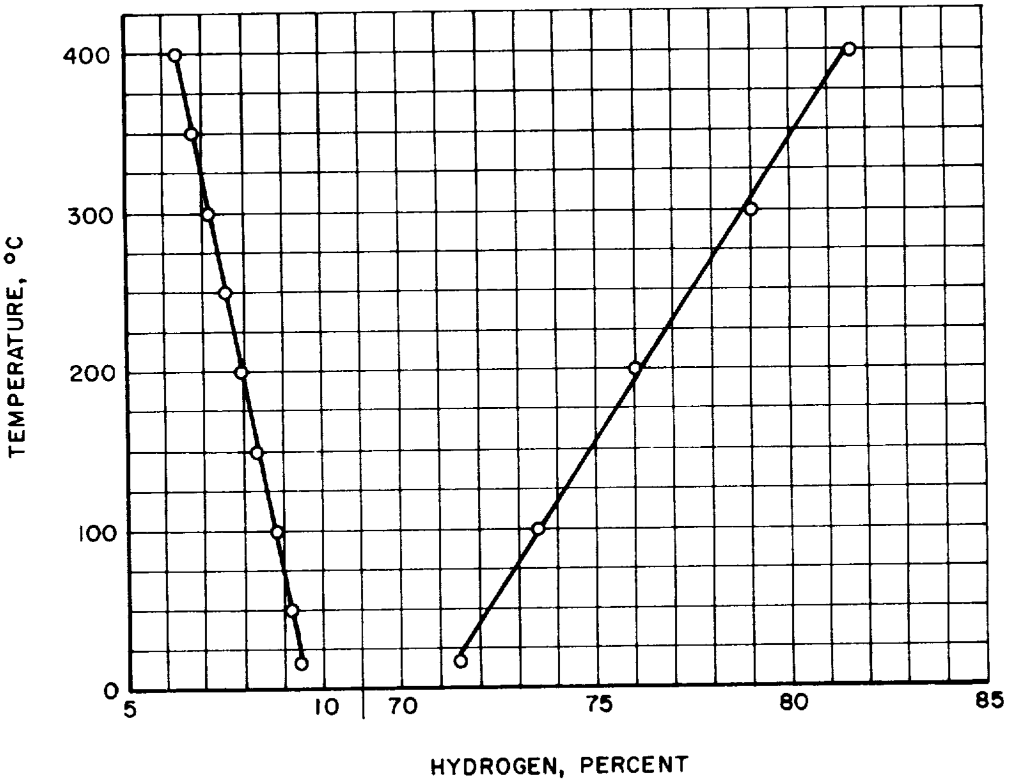

Figure 6. Influence of Temperature on Limits of Flammability of Hydrogen in Air, Downward Propagation of Flame

Section III. IGNITION OF FLAMMABLE MIXTURES

The presence of a flammable or explosive mixture does not constitute a hazard by itself, unless the possibility of a spark or other initiating source also exists. Three general types of initiating sources can be considered potentially hazardous: electrical sources, hot surfaces, and hot gases.

1. Electrical Sources

a. Electrostatic Sparks

Arthur D. Little[10] conducted studies which lead to the conclusion that significant electrostatic charges can be generated in well-grounded systems during storage and handling of liquid hydrogen. Because of the extremely low electrical conductivity of hydrogen, these charges persist over long periods of time in storage. The relaxation time for the liquid is at least one thousand times that of jet fuels.

The most serious condition for buildup of electrostatic charges would exist during two-phase flow in transfer operations. The circumstances would be particularly critical during initial cooldown of the system when both phases are present in the connecting pipelines.

Test results established that static charge concentrations generated by the liquid are much less severe since, in the streaming test apparatus, the voltages measured during liquid flow were less than 10 percent of those recorded during periods of two-phase flow. The results do not prove whether charges were built up during the transfer of gaseous hydrogen, because in all tests two-phase flow was involved in vaporizing the liquid. However, theory suggests that no gas will generate significant electrostatic charges unless liquid or solid particles are entrained.

The level of charge buildup in all tests was insufficient to cause spark ignition. The maximum field strength generated by flow through pipelines was in the order of 10 volts per centimeter. The breakdown potential of hydrogen, however, is normally 17,500 volts per centimeter. Although under some conditions sparks or corona discharges of sufficient energy for ignition could be produced at lower field strengths than 17,500 volts per centimeter, those measured in the liquid hydrogen tests are less by three orders of magnitude. It should be emphasized, on the other hand, that more critical flow conditions than produced in the tests might lead to greater charge generation and that impingement of charged hydrogen upon an electrically insulated accumulator could give potentials that would be dangerous. Therefore, positive and reliable grounding of all components is needed in hydrogen transfer systems.

b. Break Sparks[11]

When an electrical switch is opened or when a current-carrying wire is broken mechanically, an arc may occur between the separating conductors and ignition may develop if the break occurs within a combustible gas mixture. Most of the electrical energy is converted to heat while the separating conductors are still very close together. Therefore, the ignition resembles electrostatic spark ignition at very short values of gap length and the requisite energy is in the range of millijoules rather than fractions of a millijoule. Because of the inductance of the system and because the wires represent heat sinks of rather undetermined magnitude, the exact energy dissipated within the gap cannot be specified precisely.

2. Hot Surfaces[11]

a. Small Wires

If the heating current occurs as a pulse of short duration and if the heated wire is sufficiently fine, the problem of ignition or nonignition reduces to one of energy release as in other localized sources. In most practical problems the duration of heating is fairly long (measured in seconds rather than microseconds) and the heated wire is normally considered to be a hot surface.

b. Large Surfaces

The temperature required to ignite a large volume of a flammable mixture has been found to decrease with increase in wire diameter. This general behavior is illustrated in Figure 7. Since a heated wire sets up convection currents in a flammable gas situated in a gravitational field, the contact time between the heated surface and a small adjacent volume of gas is limited. However, increasing the wire diameter tends to increase the contact time and, therefore, decrease the surface temperature required for ignition. An additional decrease in the surface temperature is obtained when the flammable mixture is surrounded with a relatively large heated container in place of the heated wire or when the gravitational field strength is reduced, thereby increasing the contact time between the flammable mixture and the heated surface.

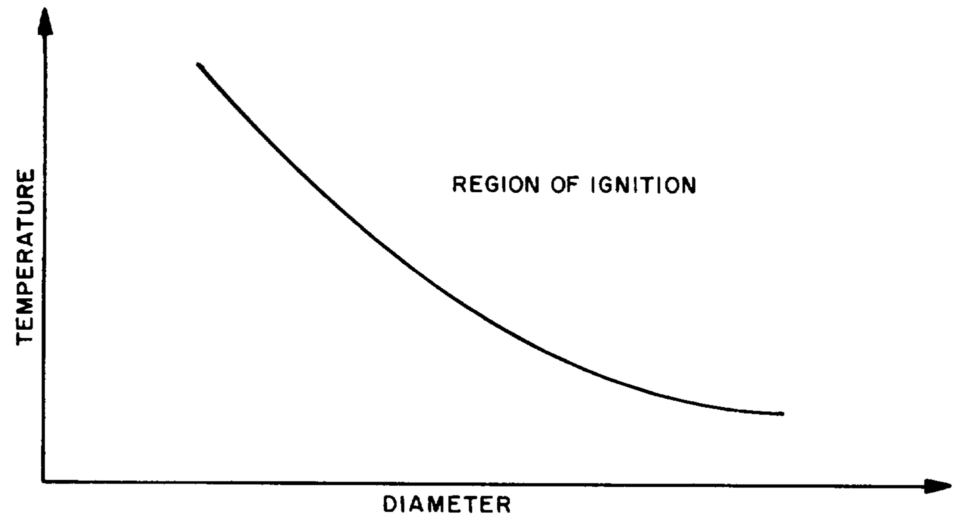

Figure 7. Effect of Wire Diameter on the Ignition Temperature of a Flammable Mixture

3. Hot Gases in the Absence of Surfaces[11]

Pilot flames of sufficiently small size may be treated as point sources, and the critical energy flux is predictable from spark ignition data. However, in most cases, pilot flames are extended sources and the critical factor for ignition is the temperature of their combustion products.

Ignition by streams of hot gases has been a specialized subject developed mainly in connection with hazards of flammable atmospheres in coal mines and in the design of engines wherein flames may be stabilized by recirculating combustion products.

Section IV. DETECTION OF FIRE AND EXPLOSION HAZARDS

Parametrics, Incorporated,[12,13] developed a hazardous gas detector which is capable of detecting not only hydrogen, but also fluorine, chlorine trifluoride, and oxygen difluoride. The system involves the techniques of radiochemical exchange using kryptonates.[12] A kryptonated solid undergoing a chemical reaction releases krypton-85 because of the chemical destruction of the surface layers. The amount of activity released is proportional to the agent with which the kryptonated solid is reacting. Conceivably any solid material may be selected which displays good reaction characteristics with the constituent to be detected and its radiokrypton homolog applied as the means of measurement. Almost any reaction can be made to release radiokrypton as an easily counted gas.

The fabricated model based on this technique was shown to be capable of producing a reliable warning indication when concentrations of hydrogen exceed 0.5 or 1.0 percent. The instrument developed under this program is relatively small and compact, simple to operate, and reliable. Nevertheless, recent advances in the preparation of kryptonated sensors would allow still further simplifications in the design with consequent decrease in size and weight, increase in reliability, and reduction in maintenance requirements.

The experimental apparatus devised for the investigation of kryptonated sources as possible detection systems for hydrogen is shown in Figure 8. The essential element of this system is the reaction vessel. The vessel is a quartz oven with a shelf for mounting the source and an aluminum window for counting the source with the Geiger tube. The vessel can be maintained at any temperature by controlling the power through nichrome heating coils encompassing the vessel. The temperature of the source can be constantly monitored by means of a thermocouple placed on the shelf adjacent to the source.

Various concentrations of hydrogen were passed over a kryptonated source. The hydrogen concentration was varied by diluting one-percent hydrogen in nitrogen mixture with pure nitrogen or air. The loss in activity of the source was determined by means of the Geiger tube and recorded. The loss in activity was determined at known concentrations of hydrogen and known source temperatures.

Analytical grade copper oxide wire was kryptonated using the high pressure, high temperature technique. Approximately three milligrams of copper oxide (CuO) wire was placed on the shelf of the reaction vessel.

The reaction vessel was heated to a constant temperature while pure nitrogen was passed over the source. The rate of decrease of the source was determined using a Geiger tube as a detector. The readout mechanism consisted of an R. E. A. C. Model H580 ratemeter and a Leeds and Northrup Model S recorder. During the heating cycle, kryptonated CuO degasses until a constant temperature is reached. Once the source has been stabilized at a constant temperature, activity will be released only if a chemical or physical reaction at the surface occurs. Thus by passing hydrogen over a kryptonated copper oxide source maintained at a constant temperature, reduction will occur with a resultant decrease in the source activity.

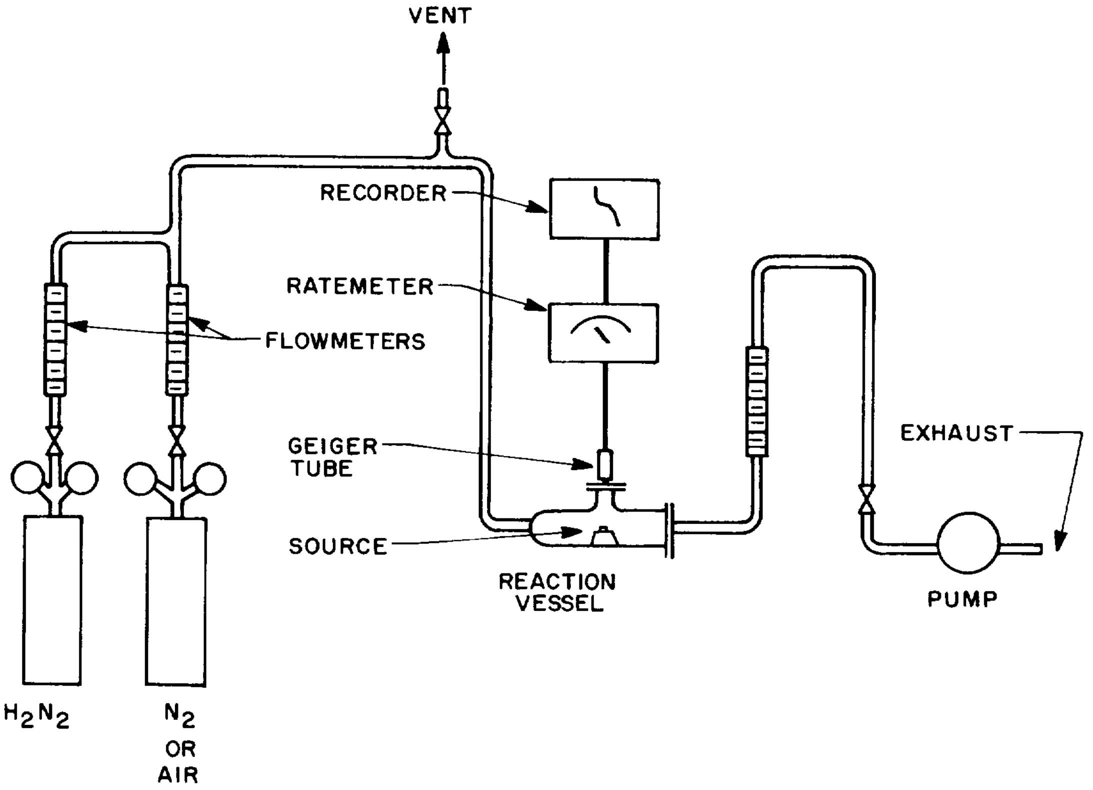

Figure 8. Hydrogen Detection System

A series of runs using kryptonated copper oxide under varying conditions of temperature and hydrogen concentration were made. It was found that as the hydrogen concentration changes, the rate of decrease of the source changes. This rate of decrease can be used as a measure of the hydrogen concentration. The percentage change per minute at any specific concentration can be found by normalizing the count rate and determining the percentage loss in t minutes. This calculation is shown in Table II. The source, S, in this table is expressed in counts per minute (cpm).

Table II. Response of Kryptonated Copper Oxide to Hydrogen

% H_2 Concentration	S_0 cpm at t = 0	S cpm at t	$\left[\frac{S}{S_0}\right]_{t=0}$	$\left[\frac{S}{S_0}\right]_t$	t (min.)	$\frac{d(S/S_0)}{dt}$ % loss/min.
0.17	44×10^3	38×10^3	1.0	0.863	21	0.652
0.33	38×10^3	25×10^3	1.0	0.657	16	2.14
0.49	25×10^3	16.5×10^3	1.0	0.660	9	3.75
1.00	16.5×10^3	5.1×10^3	1.0	0.309	9	7.68

The rate of reduction is in general a function of temperature and hydrogen pressure. It can be expected that the higher the temperature and concentration the faster the rate of reaction. Since the rate of reaction is directly proportional to the percentage decrease in activity per minute of a kryptonated oxide, this value can be expected also to increase with increasing temperature and hydrogen concentration. Figure 9 is a plot of the percentage in activity lost per minute from a kryptonated CuO source at 600° and 650° C. At both temperatures the slope of the line is constant and equal to 3/2, indicating that the percent decrease is proportional to the 3/2 power of the hydrogen concentration.

This can be mathematically written as

$$\frac{d(S/S_0)}{dt} = -K\left[H_2\right]^{3/2}$$

where K is a function of the temperature.

Figure 10 is a plot of the rate of decrease of the source versus the reciprocal temperature on semilog paper. This plot shows an Arrhenius temperature dependency, i.e., ln $d(S/S_0)/dt$ is linear with $1/T$. From the Arrhenius temperature dependency, it is possible to derive an equation describing the reduction process. From Figure 10, ln $d(S/S_0)/dt$ is proportional to $-B/T$ where B is a constant dependent on the activation energy, i.e., $B=E/R$, E is the energy of activation, and R is the gas-law constant.

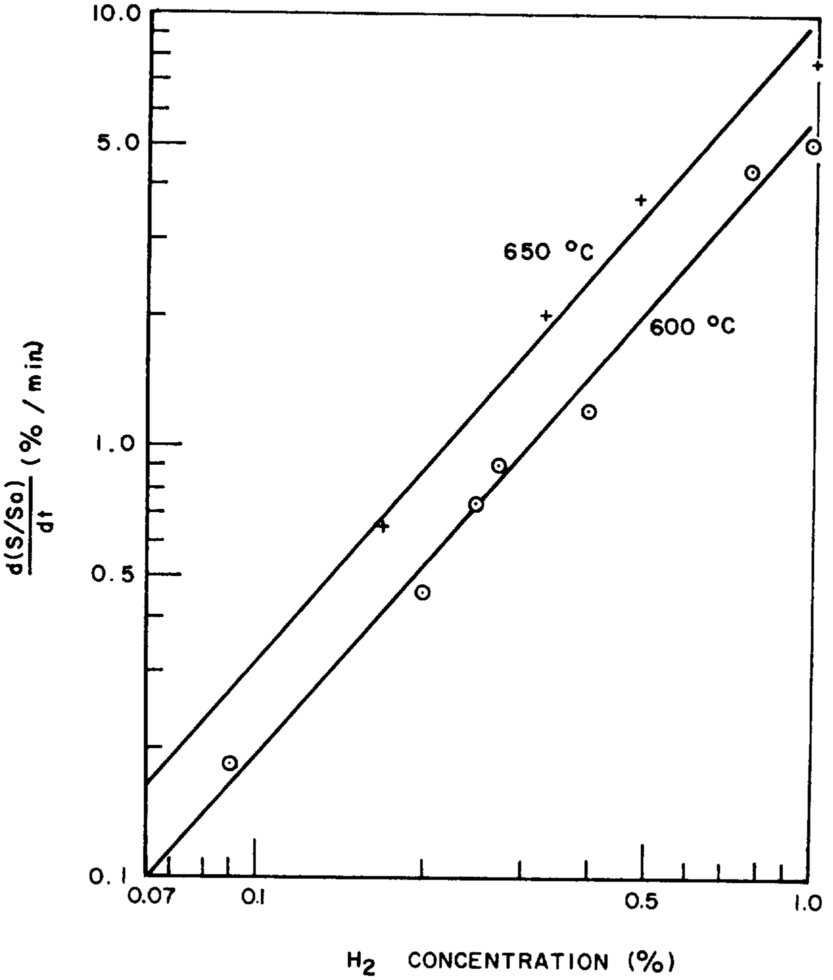

Figure 9. Response of CuO (Kr^{85}) to Hydrogen (N_2 Atmosphere)

Then,

$$\frac{d(S/S_O)}{dt} = -Ce^{-E/RT}$$

where C is a function of $[H_2]^{3/2}$ and at any constant concentration is constant, i. e., $C = k[H_2]^{3/2}$. The complete equation then is

$$\frac{d(S/S_O)}{dt} = -k[H_2]^{3/2} e^{-E/RT} .$$

From the slope of the Arrhenius plot E/R is found to be 9.05×10^3 °K, and hence E = 18.1 kilocalories. The final equation then is

$$\frac{d(S/S_o)}{dt} = -k \left[H_2\right]^{3/2} e^{-18.1\ \mathrm{k\ cal}/RT} .$$

At 600° C, $k = 1.7 \times 10^5$ % loss/min/ $\left[\%H_2\right]^{3/2}$ determined where $\ln \left[H_2\right] = 0$, i.e., when $\left[H_2\right] = 1\%$. This equation can be used to predict the percent of activity loss from a kryptonated copper oxide source at any hydrogen concentration and source temperature.

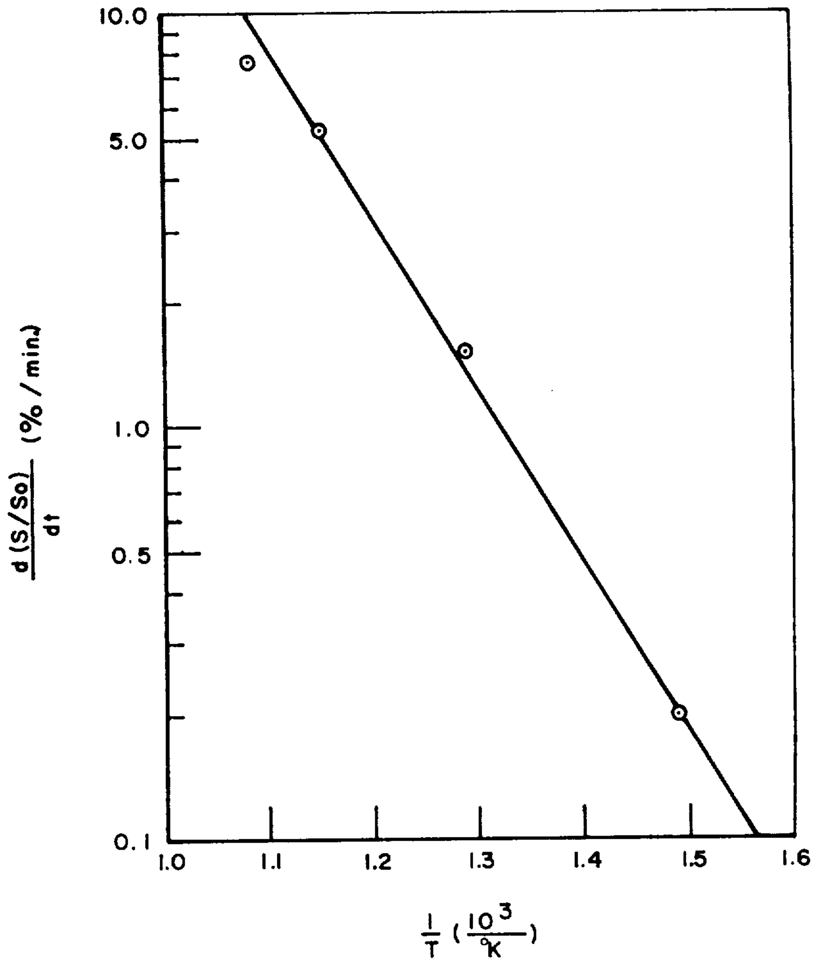

Figure 10. Arrhenius Temperature Dependency Cu0 (Kr^{85})

The results indicated in the preceding paragraphs were obtained with hydrogen diluted with nitrogen. When hydrogen was diluted with air, the results were negative. There was no release of activity when a hydrogen-air mixture was passed over kryptonated copper oxide at any temperature. It is apparent that the hydrogen must be reacting

Table III. Performance Characteristics

Characteristic	Design Specification	Measured Characteristic
A. Ambient Pressure	760 mm to 10^{-8} mm of Hg	Non-operating: 760 mm Hg to 10^{-6} mm Hg; no damage or other adverse effects. 10^{-6} mm Hg to 10^{-8} mm Hg; not measured. No adverse effects expected. Operating: 760 mm Hg to 10^{-3} mm Hg; performance as specified below. No degradation due to pressure. 10^{-3} mm Hg to 10^{-6} mm Hg; no useful performance with present sensor elements. No equipment degradation due to attempted operation at low pressure. 10^{-6} mm Hg to 10^{-8} mm Hg; not measured. No adverse effects expected.
B. Ambient Temperature	+60 to -150° Celsius	Non-operating: -20°C to 75°C; no damage. -150°C to -20°C; not tested. No adverse effects expected. Operating: -20°C to +75°C; normal operation. -150°C to -20°C; not tested. Possible difficulty of dc-dc converter start after cold soak at low temperature. Once started, no difficulties anticipated for vacuum environment.

with oxygen on the surface of the copper oxide to form water vapor. In this instance no reduction of the oxide occurs, and hence no loss in activity is observed. Kryptonated copper oxide can be used as a hydrogen detector only in an atmosphere void of oxygen. However, it was discovered that a one-to-one mixture of kryptonated platinum oxide (PtO_2) and inert molybdenum trioxide (MoO_3) gives excellent sensitivity with hydrogen in an air atmosphere. It is believed that the mechanism is one of diffusion of the hydrogen through the MoO_3 and into the PtO_2, where reduction occurs. The MoO_3 acts as a coating, which inhibits the reaction of hydrogen with oxygen, allowing the hydrogen to diffuse through its surface.

Several other materials were investigated by Parametrics, Incorporated[14] for their sensitivity in the detection of hydrogen in air. The optimum host material was found to be a 50:50 mixture of kryptonated platinum dioxide and aluminum oxide. The sensitivity obtained was 125 percent per minute for a one-percent hydrogen concentration at the maximum experimental temperature of 435° C.

The Bendix Corporation, [15] Research Laboratories Division, Southfield, Michigan,* designed a compact miniature area hydrogen detector suitable for use in the unpressurized sections of large rocket booster and sustainer stages. The hydrogen sensor element consists of a thin palladium film deposited on a gas substrate. When exposed to hydrogen gas, the electrical resistance value of the element increases. The magnitude of the change is dependent upon several factors, such as hydrogen partial pressure, the film temperature, and the film resistance. An effort was made to design this detector to minimize the effect of these factors except the hydrogen partial pressure. Thus, a change in the sensor element electrical resistance is directly attributable to the hydrogen partial pressure at the surface of the element. The characteristics of this detection system are summarized in Table III.

---

*This work was conducted for the George C. Marshall Space Flight Center, Huntsville, Alabama, under Contract No. NAS8-5282.

Table III. (Continued)

Characteristic	Design Specification	Measured Characteristic
C. Vibration	0-2000 cps; 35 g's Random noise, 15 minutes in each of three mutually perpendicular planes	Not tested. Difficulties not expected for electronic control assembly. Possibility of blower motor bearing failure in sensor assembly.
D. Sensitivity	Ten parts per million of hydrogen in air	Sensor element is responsive to hydrogen partial pressure not concentration ratio. Sensitivity limit is about 10^{-3} Torr. Hydrogen concentration limit is about one part per million in non-oxidizing gases at STP, 1000 parts per million in air at STP.
E. Accuracy	Five percent of actual gas concentration	Ten percent of indicated hydrogen pressure plus oxygen offset, which is equivalent to between 0.01% and 0.5% of oxygen pressure.
F. Range	Ten parts per million to 5000 parts per million (wide range is desirable)	Equipment range set for one part per million to 5000 parts per million at STP. (Essentially a square root scale to extend response indication.)
G. Output Signal	Analog output proportional to quantity of hydrogen (0-5 volts dc desired)	Zero-5 volts dc with ground system isolation. D-C signal is proportional to hydrogen pressure raised to the 0.53 power; desired for extended range.
H. Response Time	500 milliseconds or better	500 milliseconds or slower; response time, (0-10%), is inversely proportional to square root of hydrogen pressure.

Table III. (Continued)

Characteristics	Design Specification	Measured Characteristic
I. Stability	The system must be capable of continuous operation and must maintain calibration curve to ± 1% for eight hours without adjustment	The equipment is capable of continuous operation and will typically remain calibrated to within ± 20% for eight hours with present film elements.
J. Power Available	28 volts dc	Requires 28 ± 5 volts dc.
K. Physical Dimensions	The latest miniaturization techniques should be used to produce a compact, explosion proof package	Control assembly is a 4 in. x 2.5 in. x 3 in. box on a 3.00 in. x 5.31 in. base. Sensor assembly is 1.6 in. x 1.0 in. x 1.0 in. mounted on a 1.00 in. x 2.24 in. base. Interconnecting cable is 1/2 in. diameter 5 ft long. Terminal board construction is employed with all hard wiring connections. Entire control assembly is internally enclosed in silicone foam resin.
L. Weight	The total system weight should not exceed 3 lb	The system weight, for the delivered sensors, each containing a control assembly, sensor assembly, and interconnecting cable, are as follows: SN 101 2 lbs 4.5 oz SN 102 2 lbs 6.75 oz SN 103 2 lbs 7.75 oz

Table III. (Concluded)

Characteristic	Design Specification	Measured Characteristic	
M. Detector Specificity	The sensor shall respond to any other gas so that output from the sensor will be an indication of the presence of hydrogen	The measured response to saturated vapors at STP were obtained for the following materials:	
		Acetone	No effect
		Benzene	No effect
		Ethyl Alcohol	No effect
		Freon, P. C.	No effect
		Methyl Alcohol	No effect
		RP1 Fuel	No effect
		Toluene	No effect
		Water	No effect
		Argon	Little effect
		Helium	No effect
		Nitrogen	No effect
		Oxygen	Interference

Thiokol Chemical Corporation,[16] Reaction Motors Division, Denville, New Jersey,* investigated the possibility of detecting hydrogen-air fires and explosions in aerospace vehicles by measuring the absolute value of radiant intensity over the oxygen-hydrogen (OH) bands of the ultraviolet and the water bands in the infrared. A surprising result was the increase in OH radiant intensity with decreasing pressure. Diffusion flame variables were hydrogen flow rate, port diameter, and pressure from 760 to less than 30 mm Hg. For explosions, mixture ratio as well as pressure were varied; the rates of increase of emission and pressure after ignition were measured to determine detection lead time. Results where possible are presented as fundamental quantities that can be applied with reasonable rigor to a variety of design situations. Detection based upon monitoring OH radiation is recommended over other properties such as infrared (IR) radiation, ionization, temperature rise, or pressure rise. The conclusions reached on the basis of this study are as follows:

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*This work was conducted under Air Force Contract No. AF 33(657)-8969 for the Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

1) In general, the monitoring of emitted radiation from the hydrogen-air flame is the best basis for detection of fires and explosions under all operating conditions for reasons of sensitivity, detection from a distance within a solid angle of view, promptness of detection, and simplicity of equipment.

2) Monitoring the ultraviolet (UV) OH emission from hydrogen-air flames is intrinsically the best way to detect hydrogen-air flames. Very small flames of a one cubic centimeter hydrogen consumption rate can be detected, and for any constant hydrogen consumption rate the radiant intensity increases as pressure decreases.

3) The water band IR emission is not a suitable property for hydrogen-air flame detection, because the radiant intensity decreases with pressure below 60 mm Hg as the radiation is purely thermal and flame temperature decreases with pressure.

4) In the case of explosions, the OH (UV) emission is the best property for detection, before vessel pressure rise to a dangerous level, over the complete range of mixture ratios tested and at all pressures where explosions may yield a dangerous pressure rise. Marginal cases exist for mixtures near the rich limit (70% hydrogen) where OH concentrations are low and the radiation levels and rates of rise are unusually small.

5) Detection of explosions through the water (IR) emission is, in one sense, easier than with the OH emission, insofar as the marginal rich mixture cases are not found. The susceptibility, however, of interference from background due to a warm environment and from atmospheric and condensate absorption render the IR approach less reliable overall than the UV.

6) A flame at one atmosphere pressure represents a more critical detection problem than at reduced pressures, because of the pressure dependence of radiant intensity and minimum flame size.

7) An explosion at one atmosphere of a near-stoichiometric hydrogen-air mixture is the most critical detection problem, because such an explosion yields the highest rate of absolute pressure rise and, thus, the minimum lead time between radiation detection and some arbitrary absolute pressure rise. However, even in these cases, a conservative lead time of about 10 milliseconds is available for a five-foot explosion propagation path.

8) The time rate of pressure rise in a compartment caused by exploding a given mixture at a given initial pressure varies with compartment geometry; for any given geometry, it is nearly inversely proportional to linear compartment dimension except during the final stages of propagation.

9) Heat abstraction from the reaction zone will decrease radiant intensity levels.

10) In any real situation, the limits of detectability of fires and explosions and the detection lead time for explosions depend upon environmental design factors such as heat sink effects, geometry, and distance of detector from the flame or ignition, and upon detection system design factors such as detector threshold, irradiance level criteria for detection, and definition of hazardous conditions. The effect of many of these factors can be extrapolated from the data reported.

11) Spectral distribution in the UV has shown small variations with pressure which, however, are not significant with regard to the detection problem.

12) Solar radiation in the UV becomes an increasing interference with altitude unless the compartment in question is closed or, if vented, light shielding of detectors is provided.

The Bureau of Mines,[17,18,19,20,21] Pittsburgh, Pennsylvania, is reviewing existing hydrogen safety practices in an effort to establish acceptable standard practices. The evaluation of several hydrogen detectors is a part of this review. The evaluation of hydrogen detectors is continuing, and some results appear in the quarterly reports published by the Bureau of Mines. Progress Report No. 6 for the period from April 1 to June 30, 1965, was the last report available at the time of this writing.[21]

A brief description of the units tested and, where possible, the results obtained are listed below. Response times were determined by exposing each detector head to a series of uniform hydrogen-air mixtures contained in a vertical two-inch diameter pipe. This pipe was equipped with gas inlet and outlet ports and a horizontal sliding plate that was actuated by a rapidly acting solenoid. The detector heads were placed in a second two-inch pipe located immediately above the horizontal plate. In practice, the horizontal sliding plate was closed and the lower chamber filled with a hydrogen-air mixture at a flow rate of 100 cm^3/sec. The response time was taken to be the time interval between the instant the solenoid was actuated and the instant the signal response reached a value of approximately two-thirds of the maximum.

Recovery times were also determined in the above described apparatus. Here, the sliding plate was removed and a hydrogen-air mixture passed through the two-inch pipe. The recovery time was taken to be the time required for a drop of approximately two-thirds in the signal response (i. e., to about one-third of the maximum) following removal of the sensing head from the two-inch pipe.

1) Unit I:[19] This instrument utilizes a sensing head remote from the analyzer system. A sample of the surrounding atmosphere is drawn by diffusion and convection through a porous cylindrical element containing two heated filaments. One filament is treated with a catalytic coating to effect a change in the resistance when the sample contains combustible gas. This change in resistance unbalances a bridge circuit in proportion to the amount of combustible gas in the sample (Table IV).

Table IV. Response and Recovery Times for Hydrogen Detectors that Sample by Diffusion

Hydrogen Concentration Volume (%)	Response Time (sec)		
	Unit I	Unit IA	Unit II
1.0	2.0	1.8	2.4
2.0	1.5	1.4	2.3
10.0	1.4	1.4	1.1
25.0	0.8		0.7
50.0	0.6	1.2	0.6
	Recovery Time (sec)		
2.0	1.0	1.0	1.7
10.0		2.1	
50.0		12.7	

2) Unit IA:[19] The analyzer assembly of this unit is of the same type as in Unit I, but the sensing element is reportedly designed for detection of hydrogen in the presence of inert gases (Table IV).

3) Unit II:[19] This instrument also uses a sensing head remote from the control and assembly. The head contains four pelletized sensing elements which are exposed to the sample gas by diffusion and

convection of the ambient atmosphere through a porous disk. Two of the pellets are treated to produce catalytic burning in the presence of a combustible gas. The resulting change in resistance of the two pellets serves to unbalance a bridge circuit which produces the required signal (Table IV).

4) Unit III:[19] This is a portable instrument. The sensing element, which contains a catalytic filament, is an integral part of the complete assembly. Samples of the atmosphere are drawn through a probe by an aspirator bulb.

5) Unit IV:[19] This unit is designed for gas analysis in the laboratory. It contains two thermal conductivity cells, which are part of a bridge circuit. In the experiments conducted by the Bureau of Mines, the sample gas flowed through one cell and the reference gas (air or helium) was confined in the other.

6) Unit IVA:[19] This is an experimental unit constructed by the Bureau of Mines. Its special feature is the thermal conductivity cells made from a single metal block bored to contain two 1.5-cubic centimeter chambers. Each chamber was fitted with a 1.5-millimeter tungsten wire positioned directly in the path of the flowing gas sample.

7) Unit V:[19] This is a portable optical interferometer containing two chambers. Samples of the atmosphere are drawn through one chamber by an aspirator bulb and air (or the standard gas) remains quiescent in the second (reference) chamber.

8) Unit VI:[19] This is a hand-held device designed to detect combustible gas leaks. A self-contained pump aspirates the sample continuously at about six cubic centimeters per second through a sensing element where catalytic combustion occurs. The presence of a combustible gas is noted simultaneously by meter response and by the cut-off of an audible tone signal.

9) Units HD-7 and HD-8:[20] Two commercial leak detectors based on monitoring ultrasonic frequencies resulting from gas escaping from a small orifice were evaluated. Since the principle behind these instruments permits them to detect sound frequencies of about 40 kilocycles from any source, they are not sensitive to hydrogen leaks only. Leaks of any gas, or indeed any source of ultrasound, may produce a response. For example, instrument HD-7 responds to sounds produced by the rustling of paper or rubbing of hands. Such poor discrimination between sources of sounds limits the unit to the detection of hydrogen leaks in an otherwise silent environment. The maximum

distance between leak and instrument, at which range a hydrogen leak can be detected, was determined by means of hydrogen leaks from a closed vessel two inches in diameter and 10 inches long. Orifices of 0.001-, 0.003-, and 0.006-inch diameter were located at one end of the axis of the vessel, which was kept at constant pressure. Orientation of the orifice was varied so that its axis was aligned with the axis of detector and facing it (0 degrees), perpendicular to it (90 degrees), or facing opposite to it (180 degrees). Unit HD-7, designed for direct aural observation, was modified by connecting a sensitive AC voltmeter to the audio output. Unit HD-8 had a built-in meter, which was used. Results of the tests are summarized in Table V. The data indicate that units HD-7 and HD-8 are potentially useful, especially for detecting small leaks from orifices pointed at the detectors.[20]

10) Unit HD-9:[21] This is a portable unit utilizing a sensing head connected to the analyzer system by a coiled cable several feet long. The surrounding atmosphere is sampled by diffusion and convection through a porous cylindrical element, which acts as a flame arrester. Two heated filaments within the porous element are balanced in resistance when the gas sample is air. However, since one of the elements is coated with a catalytic substance, the presence of hydrogen causes a rise in its temperature resulting in an unbalance in the circuit and, therefore, a readout proportional to the amount of hydrogen in the sample.

11) Unit HD-10:[21] In this portable instrument the sensing element is an integral part of the complete assembly, and the gas samples are drawn through a probe by aspiration.

12) Unit HD-11:[21] This is a console unit with a diffusion head, similar in operating principle to Unit HD-9. This unit is designed to operate at a number of stations utilizing a number of modules with one sensing head connected to each module. The units under test consisted of three sensing heads, three modules, and three sensing elements. Measurement of the output of the instrument was made with a Ballantine AC Vacuum Tube Voltmeter (Table VI).

13) Unit HD-12:[21] This is a console type instrument that samples by aspiration. The sensing element, containing catalytic filaments is remote from the rest of the assembly and is enclosed in an explosion-proof container designed for Class 1, Group B atmospheres. Samples of air are drawn through this unit, which is fitted with flame arresters both at the inlet and outlet ends. The amplifier and control unit are to be located in a safe area and are designed to indicate percent of lower explosive limit (LEL) of hydrogen (Table VII). Suitable relays are incorporated to set alarms and indicate malfunctions.

Table V. Threshold Distance for Ultrasonically Detected Leaks

Unit	Orientation of Leak	Pressure, psig	Flow Rate of Hydrogen, cfm x 10^4	Threshold Detection Distance, ft
HD-7, Orifice Diameter 0.001-in.	0°	15	25	0.08
		50	71	12
		100	180	25
	90°	15	25	Not Detected
		50	71	3
		100	180	12
	180°	15	25	Not Detected
		50	71	1.5
		100	180	3.0
HD-7, Orifice Diameter 0.003-in.	0°	1	71	0.25
		5	130	45
	90°	1	71	Not Detected
		5	130	30
		15	230	45
		30	420	55
HD-7, Orifice Diameter 0.006-in.	0°	1	230	0.25
		5	560	>60
	90°	1	230	Not Detected
		5	560	>60
	180°	1	230	Not Detected
		5	560	12
		15	950	20
HD-8, Orifice Diameter 0.003-in.	0°	1	71	5
		5	130	55
	90°	1	71	Not Detected
		5	130	30
		15	230	50

Table VI. Unit HD-11 Response and Recovery Times

Hydrogen Concentration (%)	Response Time (sec)	Recovery Time (sec)
50		10.1
15	1.4	
4	2.2	6.4
3	2.1	5.4
2	2.1	4.4
1	3.4	2.9

Table VII. Unit HD-12 Effect of Sample Flow Rates

Hydrogen Concentration (%)	Meter Reading (Percent LEL)		
	$7\ ft^3/hr$	$14\ ft^3/hr$	$28\ ft^3/hr$
1	18	24	30
2	50	50	60
3	68	70	72
4	84	100	96

Rocketdyne,[22,23] a division of North American Aviation, Inc., is investigating the technical aspects of a Saturn Vehicle Fire Detection System.* The aim of this study is to obtain data on the radiative characteristics of hydrogen fires and to apply these data to the design and development of a fire detection system for the upper stages of the Saturn vehicle. The system must discriminate against false signals from sunlight and from the rocket engine exhaust plume radiation.

The fire detection system under development is composed of three subsystems. The functional role of each is described briefly.

1) OH Flicker Detector: The basis for this subsystem is twofold. First, all flames are known to scintillate; hence, the detection of time-varying radiation provides a measure of discrimination against emitting or reflecting false signal sources. Second, the OH radical is a short lived species formed as an intermediate combustion product; hence, detection of its radiation is indicative of a fire.

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*This work is being performed under Contract No. NAS8-11656.

hydrogen concentration will give approximately a six percent change in the acoustic velocity since the acoustic velocity ratio is a function of the square root of the inverse ratio of the masses. Such a change is quite significant since a 40° C temperature change would be required to create a corresponding differential in velocity. A small acoustic interferometer could be readily constructed of roughly 20 wavelengths in arm lengths where one arm is maintained in a normal atmosphere and the second arm is in the medium under consideration. A null to null variation in signal would indicate a one percent hydrogen concentration variation with such an arm length.

The basic mechanism of any acoustic system designed to evaluate the concentration of a gaseous material (such as hydrogen in a gaseous medium) may be expressed in the following manner. At any arbitrary temperature the velocity of sound is specified by the relationship:

$$v = \left[\alpha \frac{\gamma_i RT}{M_1} + (1 - \alpha) \frac{\gamma_o RT}{M_o}\right]^{1/2} \qquad (1)$$

where v = velocity in cm/sec

α = the mole fraction of the material under consideration

$1-\alpha$ = the residual mole fraction

R = the universal gas constant $8.31 \times 10^{+7}$ erg $°K^{-1}$ grm mol wt^{-1}

M_1 = molecular weights of the constituents

γ_i = the ratios of specific heat of the gases

T = the absolute temperature at which the velocity is being measured

for $\alpha = 0,\ v = v_o = \left(\frac{\gamma_o RT}{M_o}\right)^{1/2}$.

Hydrogen has a molecular weight $M_1 = 2.016$ and normal atmospheric air has a molecular weight $M_o = 28.9$. For the purpose of this discussion, the two media may be considered diatomic so that $\gamma_1 = \gamma_o = 7/5$. Relationship (1) then may be rewritten as follows:

$$\frac{\Delta v}{v_o} = \left[\alpha\left(\frac{M_o \gamma_1}{M_1 \gamma_o} - 1\right) + 1\right]^{1/2} - 1 = \text{the fractional change} \qquad (2)$$

in velocity due to the mole fraction α of hydrogen in air is of the order

$$\frac{\Delta v}{v_o} \sim \frac{\alpha}{2}\left(\frac{M_o}{M_1} - 1\right) = 6.7\,\alpha \quad (\gamma_1 = \gamma_o) \tag{3}$$

i.e., one percent hydrogen changes the acoustic velocity by 6.7 percent.

For all normal variations in common potential atmospheric constituents the value of the molecular mass M_1 is of the order of M_o, so that on a practical basis one could consider the velocity variation due to hydrogen only. To illustrate, let us introduce the velocity variation due to the known excursion of water vapor concentration, i.e., percent of atmospheric water vapor 0.2 to 4 percent. Since this is a triatomic molecule (H_2O) $\gamma_1 \cong \frac{9}{7}$ and equation (1) may be reduced to:

$$\frac{\Delta v}{v_o} \cong \frac{\alpha}{2}\left[\frac{M_o \gamma_1}{M_1 \gamma_o} - 1\right] = 0.265\ \alpha. \tag{3a}$$

One therefore may expect a fractional velocity change of approximately one percent for the entire potential vapor density variation in H_2O. This is equivalent to a 0.15 percent variation in hydrogen concentration.

Temperature variations may also be considered on the same basis:

$$\frac{\Delta v}{v_o} = \left(\frac{T_1}{T_o}\right)^{1/2} - 1\ . \tag{3b}$$

Using this relation and assuming a standard temperature of the order of 300° K, one would require about 40° C departure from 300° K to create a fractional velocity change equivalent to that induced by one percent hydrogen concentration in the atmosphere.

One would be quite satisfied with these results if helium were not used as a flushing agent. This is the major conceivable material capable of causing difficulty for acoustic systems. To illustrate, one again uses equation 3a. For a monatomic gas $\gamma_1 = 5/3$ and the value of the fractional velocity change becomes

4) Kryptonate quenching[12]

5) Molecular resonance detection of: (a) products of major and minor atmospheric constituents with hydrogen, and (b) products of inhibitor reactions with hydrogen

6) Mass spectroscopy

7) Chromatography

8) Quenching of resonance radiation

9) Palladium tube sieve

10) Laster excited Raman spectra

11) Gamma and X-ray techniques

12) Palladium resistor

13) Fuel cell system (hydrogen electrode)

14) Acoustic techniques

15) Catalysis

A brief discussion of these systems appears in the study,[25] and it was concluded that the detection of gaseous hydrogen is best accomplished by one, or a combination, of the following systems:

1) Acoustic (partial volume sensing by gas transport phenomena)

2) Kryptonate[12]

3) Electrochemical (point sensing by fuel cell action)

The system involving the use of kryptonated solids is discussed elsewhere in this report. The other two techniques are reviewed below:

First, the electrochemical system using the fuel cell hydrogen detector is discussed. Although there are many electrochemical systems, only those cells operable at (or near) room temperature are of potential usefulness for hydrogen detection. These room-temperature cell systems, at present, may be subject to partial "drowning" in the atmospheric water vapor or in their own generated water. Future research is needed to render this technique feasible for practical hydrogen detection.

Second, the acoustic system utilizing acoustic absorption and velocity methods is discussed. If one considers the acoustic velocity of hydrogen relative to that of air, one observes that a one percent

2) Sun Discriminator: Since this system responds only to time-varying radiation, the sun is a possible false signal source only when the solar radiation is modulated by a vibrating missile member or by the atmosphere. The technique for discrimination against sunlight is the two-color method, which is basically the detection of the amplitude versus wavelength envelope by a two point (wavelengths) approximation.

3) Plume Discriminator: The most promising approach for plume discrimination is with a two-frequency technique. With this technique, the amplitude versus frequency curve of the J-2 plume radiation modulation is approximated by two frequencies. If necessary, three or more frequencies are used to improve the "curve fit." The method is analogous to the two-color method used for solar discrimination.

The Radio Corporation of America[24] advanced the concept for a leak detection technique based on the addition of a radioactive contaminant, tritium-3, to the source material and, subsequently, detection of the leading tritium-3 with a solid-state detector.

Tritium-3 (H^3) decays with a half-life of 12.3 years by emission of a beta particle of 0.0180 million electron volt (mev) energy to the stable helium-3. Concentrations as low as one part tritium-3 in 10^{16} parts of ordinary hydrogen can be detected. The biological hazards from tritium are negligible, while very light shielding is required to screen out the 0.0180 mev beta particles.

A solid-state detector is available with an active layer of one micron, which is about half the thickness needed to stop the mev beta particle. If an aperture and location are selected so that about five percent of the beta particles in a cubic centimeter of gas interact with the detector and the internal detector mechanism exhibits a five percent efficiency, 2.25×10^3 pulses per second from the detector would be observed for a cubic centimeter of hydrogen leaking from the storage tank through a seal. The background radiation level from natural radioactivity was found to be insignificant.

Simmonds Precision Products,[25] Inc., Tarrytown, New York, conducted a feasibility study of a hazard evaluation system for hydrogen for the George C. Marshall Space Flight Center. Considered in this study were the following hydrogen detection systems:

1) Ultraviolet detection

2) Magnetic resonance spectroscopy

3) Microwave detection

$$\frac{\Delta v}{v_o} \cong \frac{a}{2}\left[\frac{28.9\,(5/3)}{4\,(7/5)} - 1\right] = 3.81a\,. \tag{3c}$$

It therefore requires only approximately 1.8 percent helium concentration to produce a fractional velocity change equivalent to that induced by one percent hydrogen.

An estimate of the fractional velocity change due to departure from the ideal behavior of the gas in terms of a variation in the ambient pressure may be obtained by following the technique used by Greenspan.[26,27]

$$v_o = \left(\gamma_o \frac{RT}{M_o}\right)^{1/2} (1 + \delta) \tag{4}$$

where δ is a pressure dependent correction. This may be reduced to the form we are considering:

$$\frac{\Delta v}{v_o} = \frac{1 + \delta_1}{1 + \delta_o} - 1 = \frac{\delta_1 - \delta_o}{1 + \delta_o}\,. \tag{3d}$$

Values of δ have been computed by Greenspan for some of the common gases. Using the values for air and assuming a pressure excursion from 760 millimeters to 0 millimeter, $\delta_1 = o$ and $\delta_o = 3.74 \times 10^{-4}$; one may therefore predict $\frac{\Delta v}{v_o} = -0.04$ percent for extreme pressure variations in the velocity of sound.

Section V. SUPPRESSION OF HYDROGEN FIRES AND EXPLOSIONS

An effective detonation inhibitor is defined as one which increases the ignition energy requirements for detonation in a particular system. Consequently, it is only necessary to determine the ignition energy requirements for inhibited hydrogen-oxygen mixtures in order to quantitatively evaluate the effectiveness of a particular inhibitor. Experiments were conducted by the Bureau of Mines[28] in a 30-centimeter diameter steel sphere equipped with a means of admitting the desired gas mixtures and recording the explosion pressure at the wall. An exploding wire was used as the ignition source and a piezoelectric transducer was used to record the pressure. The energy for the ignition wire was derived from a charged condenser. In these experiments, the minimum stored energy required for initiating detonation was determined to about ±5 percent for the majority of the runs. Figure 11 shows the results of this study for a stoichiometric hydrogen-oxygen mixture containing various percentages of the inhibitors methane, methyl chloride, Freon 13B1 (bromotrifluoromethane), Freon 114B2 (1, 2-dibromotetrafluoroethane), nitrogen, oxygen, and hydrogen. A similar curve labeled vacuum shows the effect of reduced pressures on the ignition energy requirements; the abscissa in this case is the percent reduction in pressure. The figure shows that methane and methyl chloride are the best inhibitors of the materials tested.

The Atlantic Research Corporation,[29] Alexandria, Virginia, studied the suppression of detonation of gaseous hydrogen-oxygen mixtures.* Quantitative data were obtained on substances known to inhibit the high-temperature reaction between hydrogen and oxygen. Complete, three-component hydrogen-air-inhibitor diagrams were constructed for nitrogen, methane, bromine, and methyl bromide. These diagrams are shown in Figures 12 through 15. Of these inhibitors, only nitrogen and methyl bromide suppress flammability in all hydrogen-air mixtures. The minimum percentages of carbon dioxide, ethyl bromide, dichlorodifluoromethane, and carbon tetrachloride required to suppress the flammability for all hydrogen-air-additive mixtures were found from the literature to be 56, 8, less than 30, and 70, respectively.

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*This work was conducted for the Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, under Air Force Contract AF 33(616)-8110.

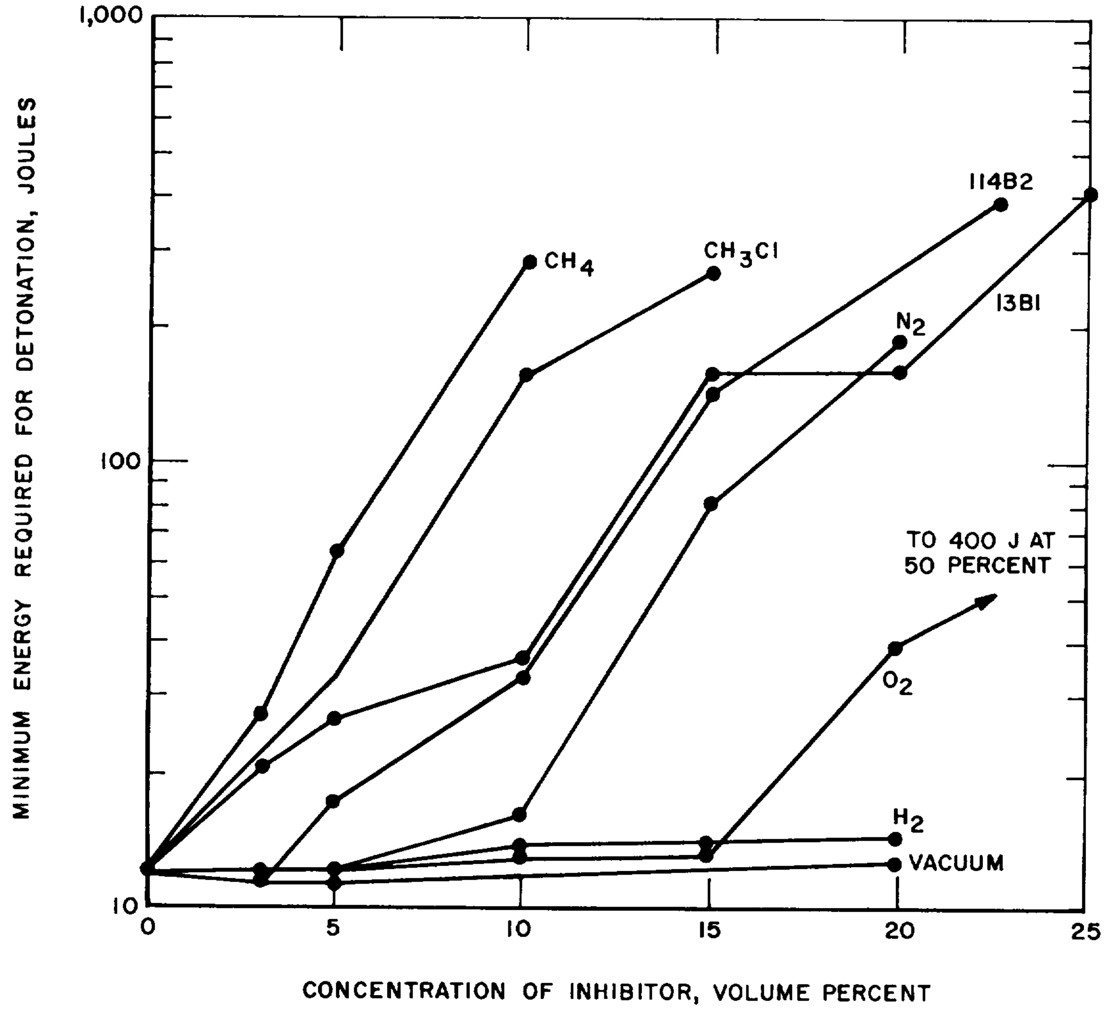

Figure 11. Minimum Stored Ignition Energies Required for Inhibition of Detonation of Stoichiometric Hydrogen-Oxygen Mixtures as a Function of Inhibitor Concentration (Volume Percent)

The Atlantic Research Corporation also studied the initiating energy required to produce detonation in gaseous hydrogen-oxygen mixtures. A total of ten additives (nitrogen, methane, methyl chloride, methyl bromide, methyl iodide, carbon tetrachloride, bromine, iron pentacarbonyl, tetramethyl lead, and water) were evaluated individually as detonation suppressants. Of these additives, methane and methyl chloride had an appreciable inhibiting effect. Iron pentacarbonyl was the best inhibitor. When 2.2 percent of this additive was employed, a detonation could not be initiated with 47 joules, four times the normal initiating energy. Although halogen-containing compounds have been reported to be fairly efficient flame inhibitors, they did not, in general, appear to inhibit initiation of detonation. The three bromine-containing inhibitors (Br_2, HBr, and CH_3Br) were about as effective as nitrogen. Methyl iodide was intermediate between the bromide and the chloride. Carbon tetrachloride, on the other hand, was found to be quite an effective sensitizer.

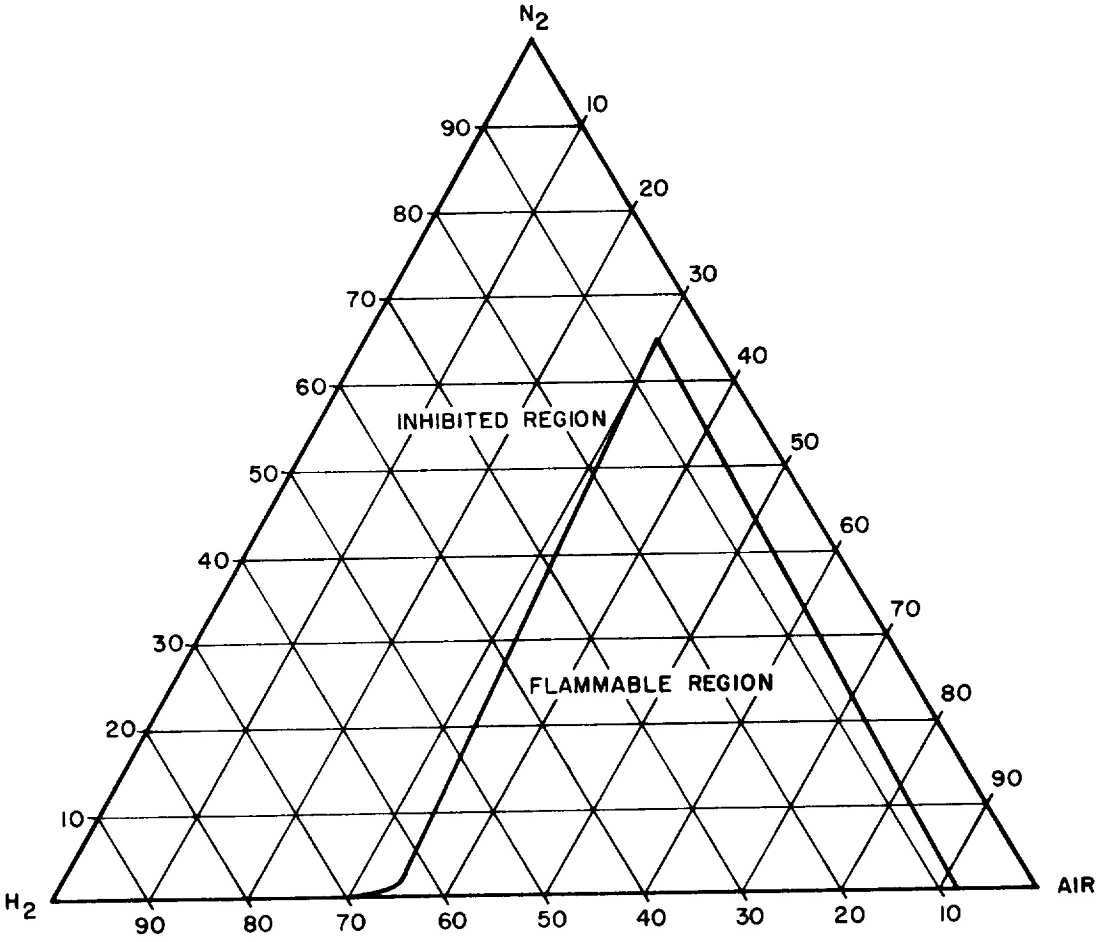

Figure 12. Flammability Limits: Hydrogen-Air-Nitrogen System

The effect of several inhibitors on hydrogen-air flame speeds was studied* by Miller, Evers, and Skinner.[30] The measurements obtained by these investigators should be accurate within ±10 percent. The concentrations of gas mixtures are expressed as the equivalence ratio, which is defined as follows:

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*This study was sponsored by the Flight Accessories Laboratory, Aeronautical Systems Division, United States Air Force, under Contract AF 33(616)-7757.

$$\text{Equivalence ratio} = \frac{\text{actual hydrogen-air ratio}}{\text{stoichiometric hydrogen-air ratio}} .$$

An equivalence ratio of 1.0 corresponds to a stoichiometric mixture, that is, two parts hydrogen to one part oxygen. Since air contains 21 percent oxygen, the equivalence ratio of hydrogen-air mixtures can be given by

$$\text{equivalence ratio} = \frac{\text{partial pressure of hydrogen}}{0.42 \times \text{partial pressure of air}} .$$

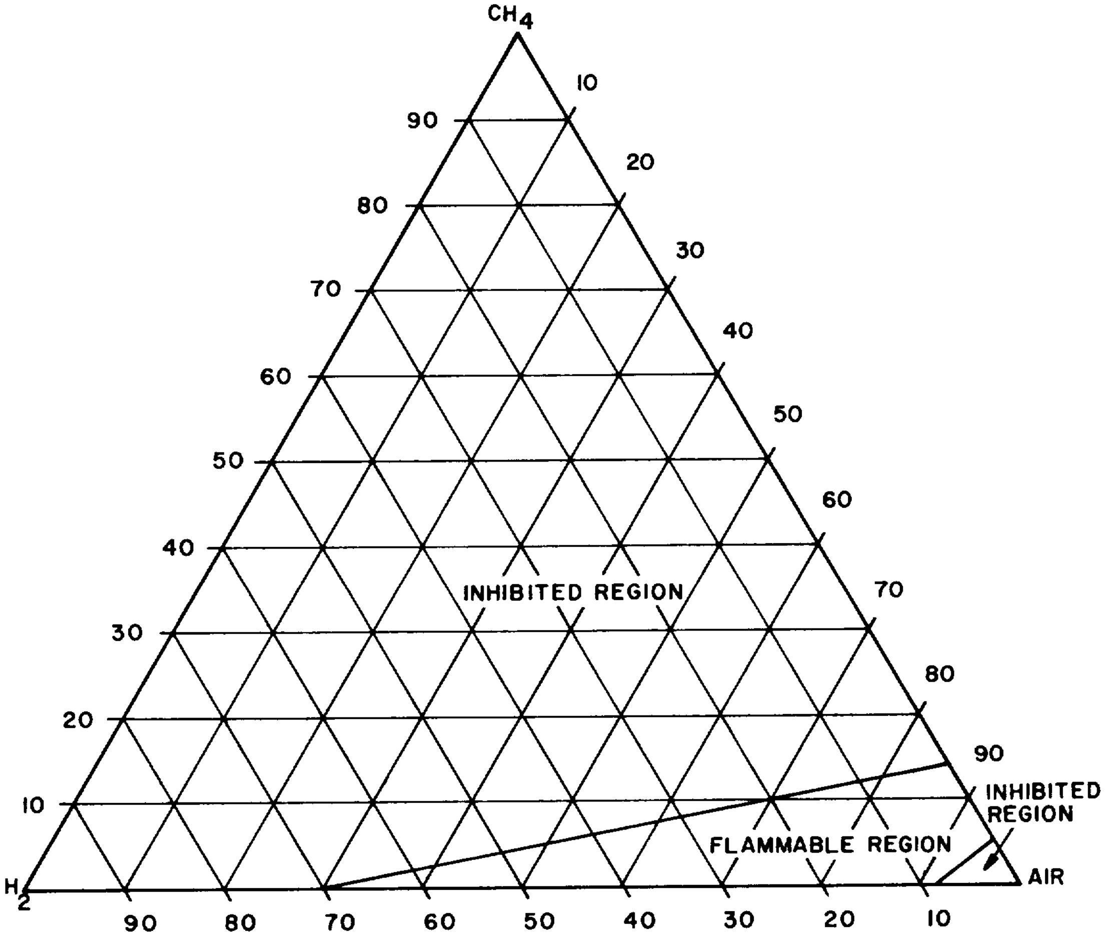

Figure 13. Flammability Limits: Hydrogen-Air-Methane System

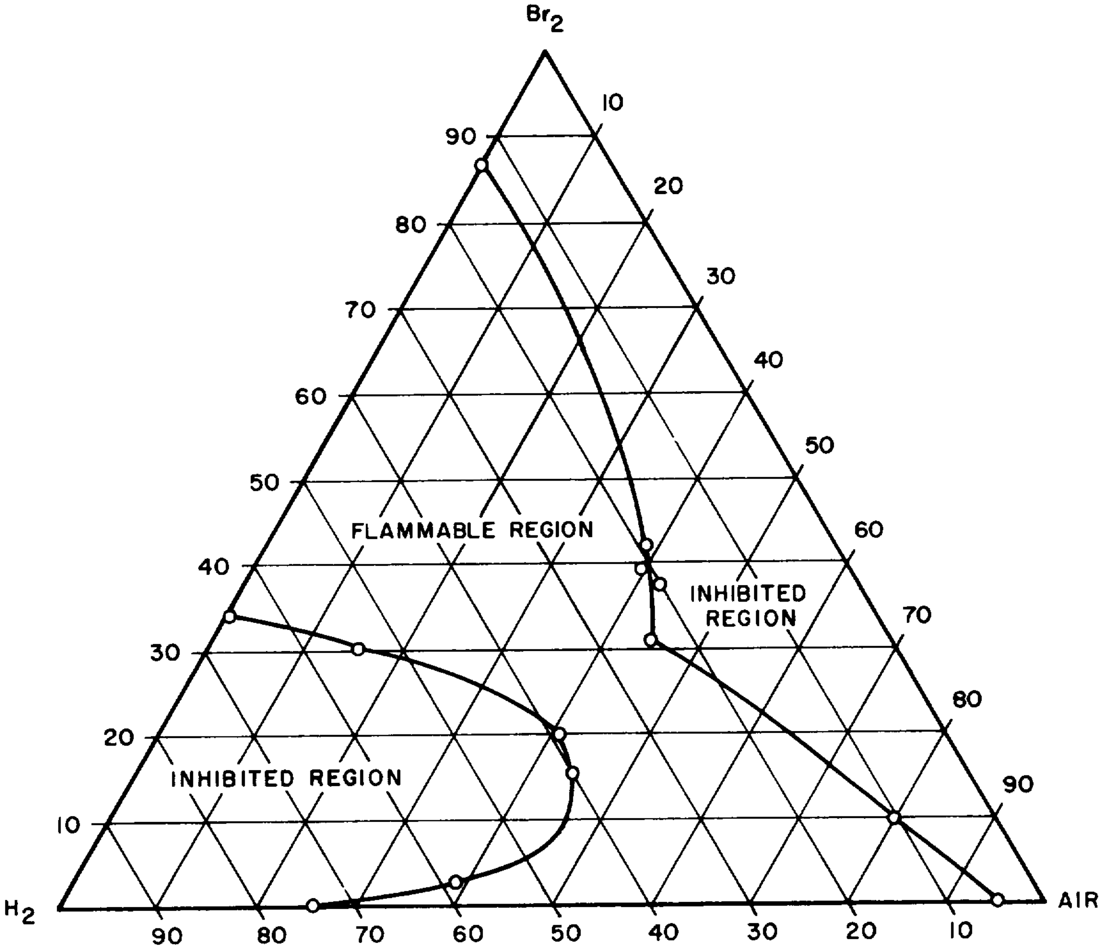

Figure 14. Flammability Limits: Hydrogen-Air-Bromine System

All experiments were carried out at one atmosphere pressure, and ideal behavior of the gases was assumed. Description of the experimental apparatus can be found in the study by Miller, Evers, and Skinner.[30] Although the effects of small amounts of 80 compounds (gases and vaporized liquids) on the flame speed were investigated, only the most promising ones are summarized here.

Figure 16 shows the results of flame speed versus equivalence ratio for six inhibitors. The concentration of the inhibitor was 0.5 percent by volume in each case. Flame speed was found to be maximum at an equivalence ratio of approximately 1.75.

Table VIII lists flame speeds for several inhibitors and compares them with flame speed when using no inhibitor in which case the flame speed was 300 centimeters per second. The gas mixture in this case consisted of an equivalence ratio of 1.75 (42.4 parts by volume hydrogen and 57.6 parts by volume air) and 2 parts by volume inhibitor. The initial gas temperature was 25° C.

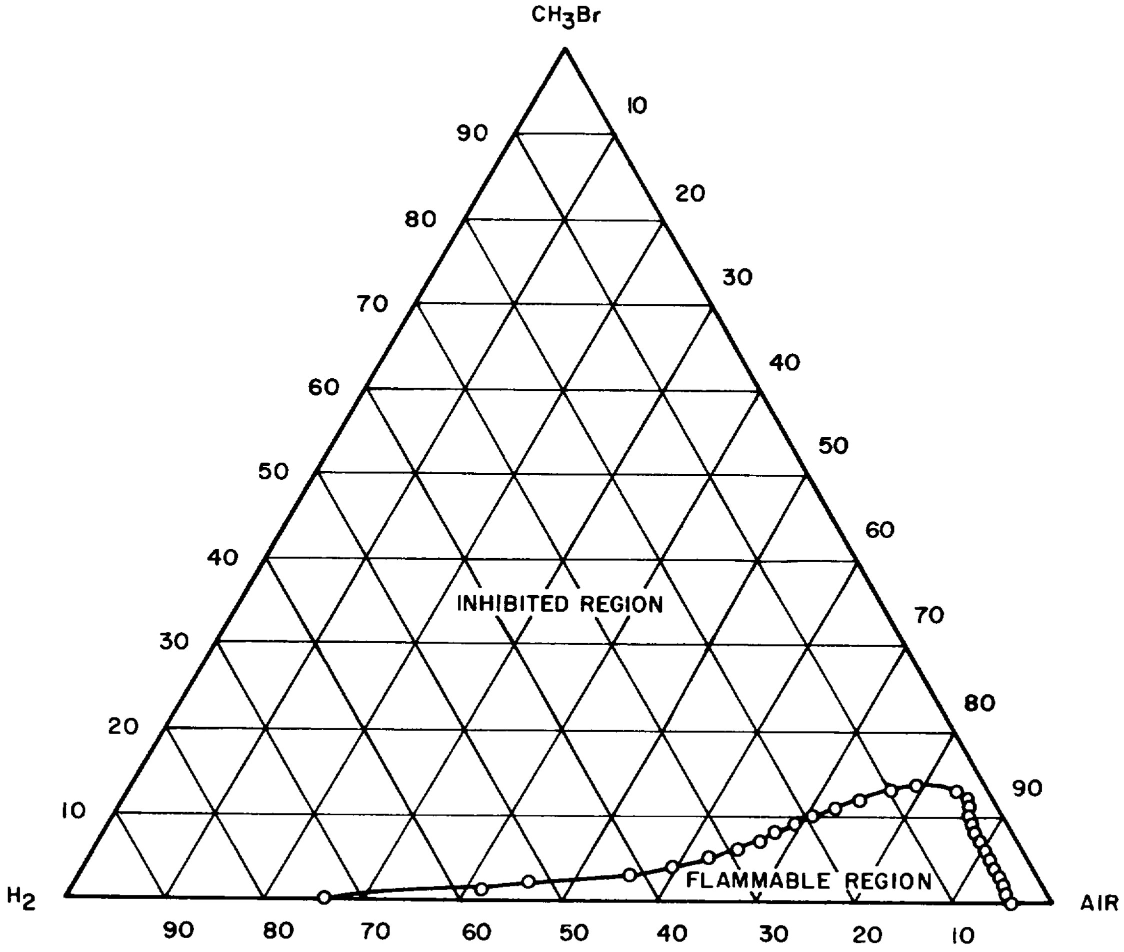

Figure 15. Flammability Limits: Hydrogen-Air-Methyl Bromide System

To determine the effect of hydrogen-air ratios on flame inhibition, a series of experiments was performed at different equivalence ratios, using several of the better inhibitors. Figure 17 shows this effect for methane at 25° C. Most of the inhibitors are more effective in rich than in lean mixtures.

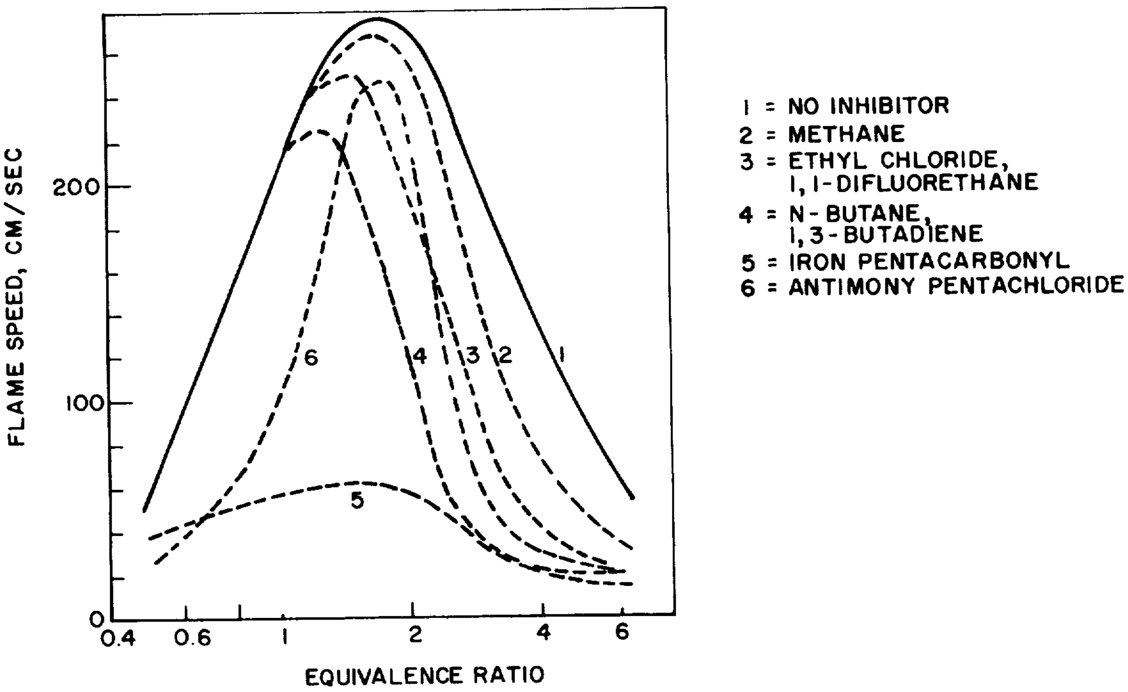

Figure 16. Flame Speed Versus Equivalence Ratio

Table VIII. Effect of Inhibitors on Flame Speed

Inhibitor	Flame Speed cm/sec
1,3-Butadiene	27
n-Butane	46
Dibromotetrafluorethane	54
Ethyl chloride	80
Ethylene	100
1,1-Difluorethane	135
Methane	144
Methyl bromide	154
Methyl chloride	199
1,1-Difluorethylene	202
Vinyl chloride	230
None	300

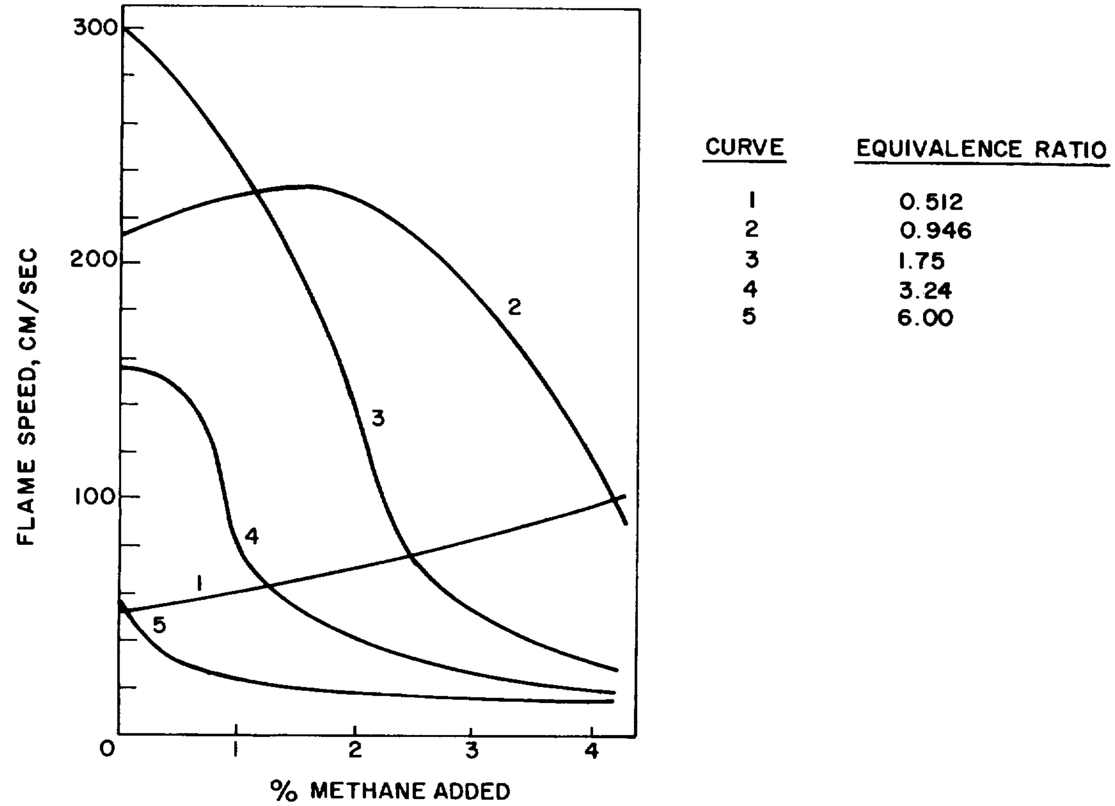

Figure 17. Flame Speeds of Hydrogen-Air-Methane Flames

The effect of temperature is shown in Figure 18, which is a plot of flame speed versus the same concentration of methane at 75° C.

Of the inhibitors investigated, the following classification can be made:

Good: Saturated and unsaturated hydrocarbons, monohalogenated hydrocarbons, brominated hydrocarbons, metal carbonyls, some metal chlorides.

Fair: Hydrogen halides, inorganic sulphur compounds, partially fluorinated and chlorinated hydrocarbons, some metal halides.

Poor: Halogens, fully fluorinated and chlorinated hydrocarbons, oxides of carbon and nitrogen, silicon and boron halides.

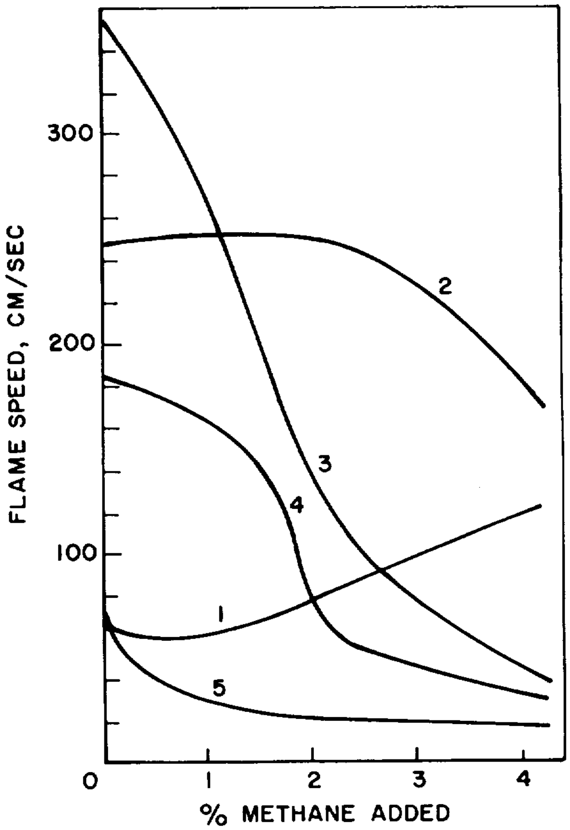

Figure 18. Flame Speeds of Hydrogen-Air-Methane Flames at 75° C

Considering the small concentrations of additives needed to produce considerable changes in flame speed, there is little doubt that these effects are due to changes in chemical kinetics, rather than in physical properties such as thermal conductivity or flame temperature.

The most likely mode of action of hydrocarbons is the removal of hydrogen atoms. For saturated hydrocarbons, this would come about by reaction of the hydrogen atoms with inhibitor to form hydrogen molecules

$$H + CH_4 \rightarrow H_2 + CH_3 .$$

In contrast, for unsaturated hydrocarbons it could be by addition of the hydrogen atoms to the double bond

$$H + C_2H_4 \rightarrow C_2H_5 .$$

In each case, a new free radical is formed, which is unstable but less reactive than the H atom removed. The hydrocarbon radicals can combine with each other; e. g. ,

$$2CH_3 \rightarrow C_2H_6 .$$

or be oxidized; e. g. ,

$$0_2 + CH_3 \rightarrow OH + HCHO .$$

The former reaction would be favored in rich mixtures, and the latter in lean mixtures. That is, in lean mixtures active species that are removed are more likely to get back into circulation, and this may be one reason why hydrocarbon inhibitors are not effective in lean mixtures.

Section VI. HYDROGEN HAZARDS TO SATURN SPACE VEHICLES

Only two production liquid hydrogen engines are in existence today, the J-2 and the RL-10, both of which are used in the Saturn space vehicles. The safety procedures that have been used for several years in the handling of liquid hydrogen are also, in general, applicable in the handling of liquid hydrogen engines. It is probably for this reason that few special procedures have been developed specifically for space vehicles. However, because of the large amounts of liquid hydrogen involved and the criticalness of space missions, considerable effort is being made to prevent hazardous situations before they occur by properly designing the vehicle. Some of the preventive measures normally applied include the following:

1) Liquid hydrogen is contained in properly designed vessels to avoid the formation of dangerous mixtures and to remove sources of ignition. This is accomplished by containing hydrogen in gas-tight systems, or diluting and sweeping away hydrogen that has leaked out inadvertently. Ignition control is applied to all potential sources of ignition, and obviously no naked lights or flames are allowed. Much attention is given to electrical devices because they can cause sparks or hot spots as a result of failure or overloading. It is common practice to place such devices in flameproof enclosures which should be sufficiently strong to withstand any explosion and to prevent transmission of the flame to surrounding flammable gas. In this country, several small electrical units, such as switches, junction boxes, etc., are certified as flameproof.

2) Prevention of flammable mixtures can also be accomplished by maintaining a positive pressure inside a container or compartment, thereby rendering leakage into it more difficult.

3) All equipment and piping is purged prior to the introduction of hydrogen. Air is eliminated by purging first with nitrogen or other inert gas.

4) Disposition of large quantities of liquid hydrogen that might be required for a vehicle test stand can be accomplished at a rate up to 10 pounds per second, with short intermittent dumps of 100 pounds per second. A disposal system developed by the Atomic Energy Commission consists of a 40-foot high stainless-steel main flare stack with a special flare tip capable of withstanding high and low temperatures, a molecular seal, propane burners, and a remote ignition system.[31]

In connection with the hydrogen hazards to space vehicles, Rocketdyne[32] is designing and developing a fire detection system for the upper stages of the Saturn vehicle under Contract No. NAS8-11656. The detection system which is being developed consists primarily of a single lens optical system, filtered to transmit only UV radiation, and a lead sulfide detector. The lens is a one-inch diameter, four-inch focal length magnesium oxide lens which transmits UV radiation at wavelengths as low as 0.25μ. The detector is actually a matched pair of lead sulfide cells in a back-to-back arrangement where one cell views the incoming radiation and the other is shielded from the radiation and is used to compensate for any change in the temperature of the apparatus. The radiometer includes provisions for a gaseous nitrogen purge to prevent deposition of contaminants on the lens.

Rocketdyne's detection system is designed to operate throughout the S-IC first stage flight, stage separation, and through second stage ignition. Consequently, it was assumed that any propellant leak and ignition source resulting from electrical and engine equipment malfunctions between the Saturn vehicle propellant loading operations and the second stage ignition, constitutes a fire-producing combination. Table IX gives the possible prime leak sources on J-2 engines as determined by Rocketdyne.

Table IX. Prime Leak Sources on Saturn Vehicle

Possible Prime Leak Sources on J-2 Engines	Leak Sources Associated with Saturn S-II Engine Compartment Installations	Possible Thermal and Electrical Ignition Sources
Turbine gas generator Liquid hydrogen turbopump Turbine gas generator crossover duct (from the LH_2 to the LOX pump turbines) The fuel pump turbine exhaust duct The gaseous hydrogen start tank relief valve	Engine service line attach panel located inside the S-II thrust cone Liquid hydrogen and liquid oxygen recirculating manifold ducts located around the thrust cone internal periphery	<u>Thermal:</u> Turbine gas generator Turbopump Turbine exhaust and crossover ducts <u>Electrical:</u> The electrical equipment and associated cabling around the Saturn S-II thrust cone periphery

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This report reviews and summarizes the hazards to aerospace vehicles caused by hydrogen. Topics such as flammability limits of hydrogen in hydrogen-oxygen and hydrogen-air mixtures, methods of detecting hazardous conditions, and means of inhibiting fires and explosions are discussed. Although relatively few references were found in the literature concerning hydrogen fire and explosion hazards in aerospace vehicles specifically, it is generally believed that many of the procedures used in the handling, storage, and transportation of liquid hydrogen in more conventional applications also apply to hydrogen used as a fuel in space vehicles.

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